VALIDATION OF IN-SITU MICROWAVE

MOISTURE METER FOR SAND

AND CONCRETE

by

Jafar Talal Allahham

A thesis submitted to the faculty of
The University of Utah
in partial fulfillment of the requirements for the degree of

Master of Science

Department of Civil and Environmental Engineering

The University of Utah

December 2017

The University of Utah Graduate School

STATEMENT OF THESIS APPROVAL

The thesis of **Jafar Talal Allahham**

has been approved by the following supervisory committee members:

Amanda Bordelon	, Chair	**03/28/2017** Date Approved
Chris Pantelides	, Member	**03/21/2017** Date Approved
Pedro Romero	, Member	**03/21/2017** Date Approved

and by **Michael Barber** , Chair/Dean of

the Department/College/School of **Civil and Environmental Engineering**

and by David B. Kieda, Dean of The Graduate School.

ABSTRACT

The moisture content in civil engineering materials determines many of the structural properties of the material such as strength and durability. In geotechnical engineering, the moisture content of soil deposits determines their susceptibility to landslides and settling. In structural engineering, the moisture content in concrete, typically measured in terms of the water-to-cement (w/cm) weight ratio, determines its compressive strength as well as other hardened properties such as permeability and shrinkage.

The moisture contents of sand and concrete composites were measured using a handheld microwave moisture meter developed for the purpose of moisture measurements in concrete. The results in concrete obtained from the meter were compared to the results obtained from the standard method of determining moisture content in concrete. In sand, the meter was able to detect the change in moisture content with a linear fit R^2 of 0.962 and 0.945 for the two types of sands tested. As for concrete, the linear fit R^2 was as low as 0.0034. The p-values obtained on concrete testing were less than the specified confidence level of 0.05, rejecting the hypothesis that the meter's average output is equal to the average actual w/cm tested. The output w/cm obtained from the meter was compared to moisture content and calculated w/cm from the AASHTO standard method. The linear fit through the data obtained from the test had an R^2 value of 0.62 or higher and a p-value of 0.91, making this method the preferred option when wanting accurate in-situ measurements.

TABLE OF CONTENTS

LIST OF TABLES

Tables

LIST OF FIGURES

Figures

ACKNOWLEDGMENTS

This research study was made possible by the funding of the Utah Department of Transportation (UDOT) and Mountain-Plains Consortium. Much appreciation is given to employees working at UDOT, Harper Precast Concrete plant, and The Point project for coordinating and allowing field batching of aggregates and tests on concrete.

I would like to thank my advisor Dr. Amanda Bordelon for guiding and mentoring me throughout my graduate academic career. I was constantly challenged to perform better and you were there to help whenever it was needed. Thank you for the opportunity to work with you and I hope one day we'll work on the same project again.

CHAPTER 1

INTRODUCTION

1.1 Motivation

The compressive strength and water-to-cement (w/cm) ratio of concrete are the two most important indicators of its performance. Typically, an increase in w/cm ratio results in a decrease in compressive strength and an increase in the porosity of the structure, both of which can cause early degradation of the structure (Kim et al. 2014). A concrete mix design often has a specified w/cm ratio and the concrete producer would make sure the concrete meets these specifications before it leaves the batch plant. However, extra water is often added after leaving the plant, or even prior to leaving the plant, such as when the truck driver washes the chute inlet to ensure all measured concrete components are mixed in the truck during transit or simply clean the chute. Water may also be added, for example, when traffic delays the concrete truck or prevents the truck from arriving at the jobsite on schedule. Water may also be added at the jobsite to improve workability or to delay setting of the mixture.

The Quality Assurance and Quality Control (QA and QC, respectively) engineers may not be able to determine whether extra water was added since the batch ticket indicating the design w/cm does not indicate if extra water was added. If the addition of excess water was not detected before the concrete was placed and hardened, the concrete may have to be demolished, which can be very costly and can increase the duration of construction. Therefore, an on-site or in-situ device to measure the w/cm of the mixture right before placing the concrete is preferred.

1.2 Determining Water Content

The in-situ water content is most commonly measured for materials like soil or wood. In geotechnical engineering, determining the water content of the soil on-site can allow for the calculation of the density of the soil and determine the level of compaction (Salgado 2008). Furthermore, knowledge of the water content is essential in determining the strength of the compacted soil and prediction of susceptibility to landslides or settling since soil consistency and settling depend mainly on its water content. In wood, the moisture content correlates to the strength of the member, as well as the likelihood of rotting which can jeopardize the integrity of the member.

Moisture meters work based on the same principal; water's electromagnetic properties differ greatly than that of solid materials used in construction such as cement powder, aggregates, soil or wood. Water is a highly polar molecule, with a dielectric constant of more than 10 times that of most rocks. Thus, a moisture meter that can measure the electrochemical properties of a mixture should theoretically be able to measure the moisture content and subsequently volume of water.

1.3 Methods for In-situ Determination of the Water Content in Concrete

There are currently limited standard methods to measure the in-situ water content of concrete. The most common and standard procedure used by QC/QA personnel today is based on the AASHTO (American Association of State and Highway Transportation Officials) T318-02 microwave oven method (AASHTO 2015). This AASHTO method relies on drying a sample of concrete in a microwave oven for at least 10 minutes and then calculating the moisture content from the difference in weight from the original wet and microwaved dry measurements. At most construction sites, concrete mixing trucks place the concrete in the desired location and leave the site usually before the results of the AASHTO test have been recorded. Thus, there is still the need for a faster method to measure the w/cm on-site.

Another method which has recently been utilized by some departments of transportation (DOTs) is to measure the moisture content of concrete is using the Cementometer™, a handheld microwave concrete moisture meter developed by the company NDT James Instruments (James Instruments 2010). This microwave moisture meter method is anticipated to be preferred over the AASHTO because it involves having a convenient handheld device which can be inserted into any concrete sample and it displays the instantaneously calculated w/cm content on the screen. The device is also anticipated to be useful for estimating moisture content in sand or other aggregates by using an approximate regression equation through the data points obtained from the unit less output value displayed on the device to back calculate the moisture content.

1.4 Research Objectives

The industry has a need for an improved and faster method to determine the in-situ w/cm of fresh concrete. A Cementometer™ device was purchased by the Utah Department of Transportation (UDOT) to be used for QC and QA testing. For this study, the main objective was to determine the accuracy and precision of the Cementometer^{TM} device for concrete mixtures of interest to UDOT. In this research, concrete mix designs from UDOT as well as custom in-house made concrete were created to calibrate the meter and validate its output. The UDOT mixtures were obtained from approved batching plants and paving sites. Furthermore, the results of the Cementometer^{TM} outputs were compared to the existing standard method for determining moisture content in concrete. A final recommendation on the applicability of the meter as a QC/QA device was made based on the calculated statistics. A summary of the objectives of this study are as follows:

1- Calibrate and then record outputs with the Cementometer^{TM} for desired concrete mix designs.

2- Perform statistical analyses on the output results obtained to determine the accuracy and precision of the Cementometer^{TM}.

3- Compare the Cementometer™ to the existing AASHTO method and give a final recommendation on the applicability of the meter.

4- Perform literature review and tests with the Cementometer^{TM} to determine the measurement sensitivity, as well as its applicability to use the Cementometer^{TM} on moisture content in sand.

CHAPTER 2

LITERATURE REVIEW

2.1 Basic Theory of Microwave Moisture Meters

2.1.1 Polarization and Dielectric Permittivity

The microwave sensor, probe, or meter operates on the principal that water is a polar molecule and has a significantly different dielectric constant (Table 1) compared to that of nonpolar materials such as cement and aggregates. A microwave-based meter is expected to be able to quantify the moisture content in a polar material.

Polar molecules have nonsymmetrical atomic geometry. If in addition to the nonsymmetry, the center of negative charges and positive charges in the bond do not lie in the same location, it can have a potential dipole moment (rotation). When an electric field is applied to polar molecules, two dipole moment effects will result. The first dipole moment is induced when the electric field aligns the charges inside the atoms, as shown in Figure 1. With a water molecule, the positive charge moves in one direction while the negative charge spins in the opposite direction; the resulting first dipole moment develops at the center of the two charges (Sands et al. 2013). The second dipole moment results due to the shift of the molecules of the material as a volume. This results in groups of molecules oriented in different directions, resulting in another moment due to the center of charges of the different groups not lying in the same location.

A dielectric constant is a measure of polarity of any material (even if the material is polar or nonpolar) when subjected to an electric field. Since water is polar, it has a high dielectric constant. Permittivity is determined as the material's resistance to an encountered electrical field, or resistance to polarization. The dielectric constant is calculated for a nonconductive material as the relative permittivity of the material with respect to the permittivity of a vacuum, as shown in Equation (1) (Nave 2012).

$$\boldsymbol{\varepsilon_r = \varepsilon_s / \varepsilon_0} \text{ (Equation 1)}$$

where:

$\boldsymbol{\varepsilon_r}$ = Permittivity relative to a vacuum (Dielectric constant)

$\boldsymbol{\varepsilon_s}$ = Measured permittivity of material in Farad per meter (F/m)

$\boldsymbol{\varepsilon_0}$ = Permittivity of a vacuum in Farad per meter (F/m)

2.1.2 Dielectric Loss

For materials that exhibit some conduction in the presence of an electric field, the energy or heat dissipated from the sample which exhibits polarization is referred to as a dielectric loss. When the dielectric material is "lossy" (high dielectric losses mainly due to conduction), the calculated relative permittivity or dielectric becomes more complex. Instead of using the simple Equation (1), the relative permittivity or dielectric constant of a material exhibiting some dielectric loss is calculated using Equation (2) (Chang 2005). An example of a material that is highly conductive is tap water, which contains ionic compounds; pure water is polar but is nonconductive and as such has lower losses. Another example of a lossy material can be concrete (Shen et al. 2016) because ionic dissolution during hydration can increase conductivity, in addition to the conductivity of the mixing water. Thus, the simultaneous measurement of the relative dielectric permittivity and loss factor is needed for an accurate estimation of the water content in fresh concrete.

$$\boldsymbol{\varepsilon}^* = \boldsymbol{\varepsilon_r} - \boldsymbol{j}\boldsymbol{\varepsilon}'' \quad \text{(Equation 2)}$$

where:

$\boldsymbol{\varepsilon}^*$ = Complex relative permittivity (Complex dielectric constant)

$\boldsymbol{\varepsilon_r}$ = Calculated dielectric permittivity constant per Equation (1) (F/m)

$\boldsymbol{\varepsilon}''$ = Imaginary dielectric loss factor (F/m)

$\boldsymbol{j}$ = $\sqrt{\boldsymbol{-1}}$

2.2 Dielectric Measurements

There are many methods involving dielectric measurements on materials at various frequency ranges. Most of the methods are considered capacitive methods because electric field is created by a capacitor system. Depending on the number of conductors used in the system, dielectric measurement methods can be classified as either transmission-line or waveguide methods (Pozar 2012). In transmission lines, the system is constructed using two conductors, as simple as two wires, and designed to propagate an electromagnetic wave between the two conductors. Waveguides are made with only one conductor, and are commonly hollow tubes, designed to transmit and confine waves inside the tube. Waveguides can be circular or rectangular (Anderson 2006). Alternative waveguide geometries also exist including a single co-axial line probe (as is used for this study at the University of Utah), or as a parallel plate capacitor design in Figure 1b.

An example of how the electric field is generated in a waveguide method can be seen in Figure 1 of a parallel plate capacitor. Electric energy is transmitted as a field through a material between the plates. The material stores some energy quantified by the relative dielectric permittivity and may also release some energy as a dielectric loss. A challenge with this parallel plate capacitor geometry is that some of the dielectric material may physically lie outside the boundary of the electric field between the plates, which can cause errors in estimating the volume of material between the plates. The probes can be encapsulated in a non-conductive material to separate them from the surroundings, creating similar conditions as a rectangular or circular waveguide. There is anticipated to be some error in measuring the dielectric of such materials due to the volume of material outside the plates.

2.3 Frequency Effect on Dielectric Measurements

The dielectric properties of a material depend on the frequency of the electromagnetic field and the temperature of the material (Chang 2005). Any electromagnetic field, regardless of its frequency, can result in some polarization of a material. However, high radio frequency microwave electromagnetic fields generally provide the more accurate material dielectric properties (Baker-Jarvis et al. 2010). A frequency within a range between 300 MHz to 300 GHz is considered a microwave frequency (Nave 2012). Dielectric losses due to conduction are also reduced at these higher microwave frequencies (Chang 2005). Shen et al. (Shen et al. 2016) stated that the ionic concentration of a solution creates a complex dielectric permittivity from conduction and is more influential at low frequencies than at high frequencies.

A network analyzer and co-axial probe owned by the Department of Electrical Engineering at the University of Utah was used to measure the dielectric constant of deionized water, dry sand, and air as a function of frequency (from 200 MHz to 400 MHz) to determine the difference in the dielectric constant between the three materials. As seen in Figure 2, the dielectric of water was as high as 76 at room temperature (21°C +/- 2°C), compared to 0.92 for oven dry sand and air. The dielectric of water and air is within a typical range. However, the measured dielectric of sand is below the reported range of 3 to 5 (Table 1). This could be due to the probe measuring mostly the air around the uncompact sand. Deionized water was selected for this measurement to minimize the effect of ions that might create dielectric conductive losses or inaccurate complex dielectric readings compared to tap water. The maximum dielectric values for deionized water was found at microwave frequencies between 280 to 300 MHz (shown in Figure 2).

2.4 Temperature Effect on Dielectric Constant

The dielectric constant of materials is affected by the temperature of the material. Typically, an increase in temperature results in a decrease in dielectric capacity of the material. This can be attributed to the energy stored as heat in the system. In water and concrete, the dielectric constant decreases linearly as the temperature is increased, as proposed by (Chen et al. 2012) and shown in Figure 3. The dielectric of water was measured over a wider range of temperatures than concrete and the linear regression R^2 of water was higher in magnitude than concrete. The higher R^2

could be from the wider range of temperatures tested and the lower variability in dielectric measurements in water than in concrete.

2.5 Pore Property Effects on the Dielectric Constant

Properties of the soil such as pore size distribution, volume, and surface area contribute to the change in the dielectric constant of soil at low and high moisture contents (Blonquist et al. 2006). Generally, the dielectric constant of soil increases with increasing moisture content. However, at low water contents, the dielectric constant is less sensitive to moisture change due to the water binding to the surface of the pores (Gadani and Vyas 2008; Wang and Schmugge 1980). Bound water is difficult to polarize. A small increase in the dielectric constant is expected as long as the water is bound to the surface. The larger the pore size, the less the surface area to volume ratio, thus more free water is available than bound water for that volume of pores. When the volume of free water increases, the dielectric constant starts increasing rapidly, as shown by Gadani et al.

2.6 Concrete Hydration Effects on Dielectric Constant

As the hydration initiates in a concrete mixture, the cement begins to dissolve into ions in the mixing and dormancy phases. At the same time, water becomes physically and chemically bound to the cement hydration products. The heat of hydration is relatively low during the first few hours but then increases significantly once the setting phase has initiated. Makul (Makul 2013) measured the dielectric constant of several concrete mixtures using an open-ended coaxial probe during the first

24 hours of hydration (including the dormancy and setting phases). In the study, Makul concluded that the initial (fresh concrete) dielectric constant is strongly affected by the initial w/cm and the heat of hydration (as varied with different cement particle fineness). Figure 4 shows the relative dielectric constant and temperature of the concrete mixture over hydration time for different w/cm mixtures. From the figure, it is apparent that w/cm has a great influence on the initial dielectric. For example, the mixture with w/cm of 0.70 had an initial dielectric of approximately 30, while a 0.38 mixture started at around 13. This is expected since the dielectric of the concrete mixture depends mainly on the dielectric of water due to its relatively high dielectric constant.

As the concrete mixtures started setting (around 6 hours), the temperature of the mixtures increases and the dielectric constants of the concrete mixtures correspondingly decrease. The dielectric constant of the three different mixtures tested decreased in time, reaching similar values for all w/cm when the concrete reached a complete setting or hardened phase (by 21-24 hours).

In addition to the dielectric permittivity decreasing as the concrete hydrates, the dielectric loss factor also decreases. For the w/cm of 0.70, the initial loss factor can be almost 30% of the dielectric permittivity (Figure 4). The loss factor decreases to zero once the concrete fully hydrates since less free ions due to the hydration process exist. The high loss factor can cause errors in measuring the dielectric of the mixture if unaccounted for, resulting in errors in measuring the volumes of mixture constituents as discussed in the following section.

2.7 Dielectric Constant of a Mixture

When the material to be tested is a mixture of more than one material, the relative dielectric constant of the composite is also more complex than as stated in Equations (1 or 2). The calculated dielectric constant of a composite mixture such as concrete is dependent on the volume of each component and dielectric constants of each component or combined component interactions.

Chen et al. proposed such an equation to calculate the net relative dielectric constant of concrete as a composite. The concrete was modelled as a three-phase material: solids (aggregates and cement), gas (air), and liquid (water). The equation was verified on fresh concrete samples with known w/cm and the measured concrete dielectric within 5 minutes of mixing using a ground penetrating radar device. The dielectric of the multiphase material was described using a Debye relaxation function shown as Equation (3).

$$\varepsilon_r = \left(\sum V_i^2 * \ \varepsilon_i + 4 \sum V_{m-1} \ * V_i \ \frac{\varepsilon_{m-1} * \varepsilon_i}{\varepsilon_{m-1} + \varepsilon_i} \right) \ [1 + (20 - T) * \alpha_c] + b$$

Equation (3)

where:

ε_r = Relative dielectric constant of composite fresh concrete.

V_i = Volumetric fraction of gas, liquid, or solid phases in the material,

ε_i = Relative dielectric constant of the specific phase,

ε_m = Relative dielectric constant of two-phase combinations (Solid+Liquid, Solid+Gas, Liquid+Gas), treated as two capacitors in series and is calculated as $\varepsilon_m = \frac{\varepsilon_{n-1} \times \varepsilon_n}{\varepsilon_{n-1} + \varepsilon_n}$ where n is the phase number

T = Temperature of the concrete mixture (°C)

α_c = Temperature coefficient of the concrete mixture (1/°C), reported as -0.04/°C

b = Calibration parameter to adjust for the homogeneity of the mix compared to discrete phase layers. Value from Chen et al. (2012) = 4.73.

While hydrating, the conductivity of concrete is expected to change due to ionic dissolution of the cement. However, since the mixtures were tested within 5 minutes of mixing, any ionic conductivity losses are assumed be low and were not studied by Chen et al. The three-phase model assumes that the layers of phases are stacked on top of each other rather than homogenously mixed. The parameter 'b' accounts for the distribution of phases instead of layered discrete phases, and was approximated by Chen et al. using a least-squares method with their data set.

Table 1 Dielectric Constant of Common Materials (Clipper Controls Inc)

Material	Minimum Dielectric	Maximum Dielectric
Water	55	88
Air	1	1
Portland Cement	5	2.6
Sand	3	5

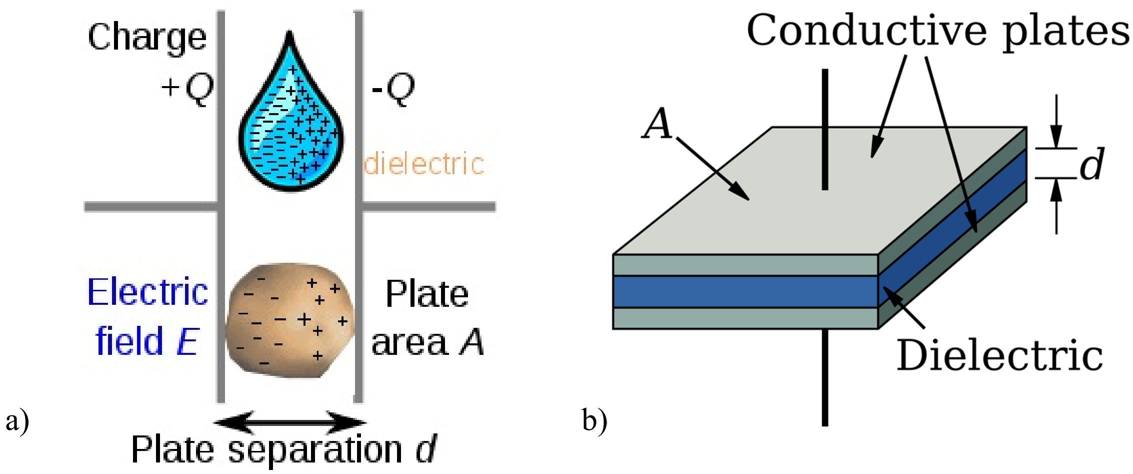


Figure 1 Parallel plate capacitor a) Polarization of dielectric materials under an applied electric field and b) Capacitor

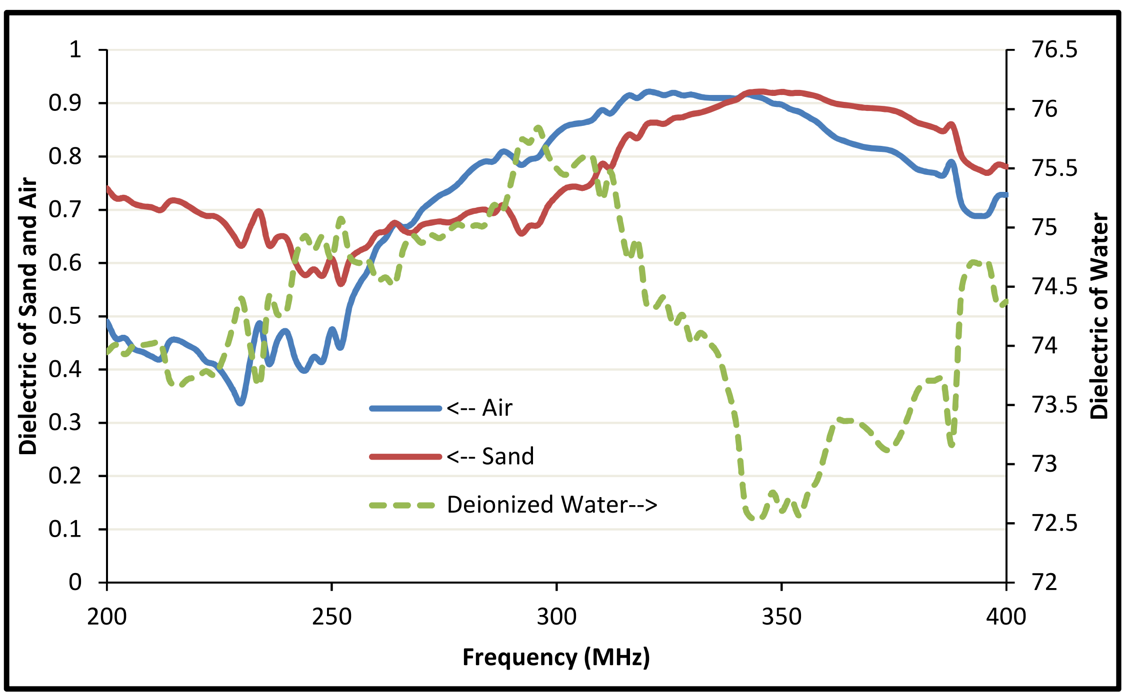


Figure 2 Measured dielectric constant versus frequency.

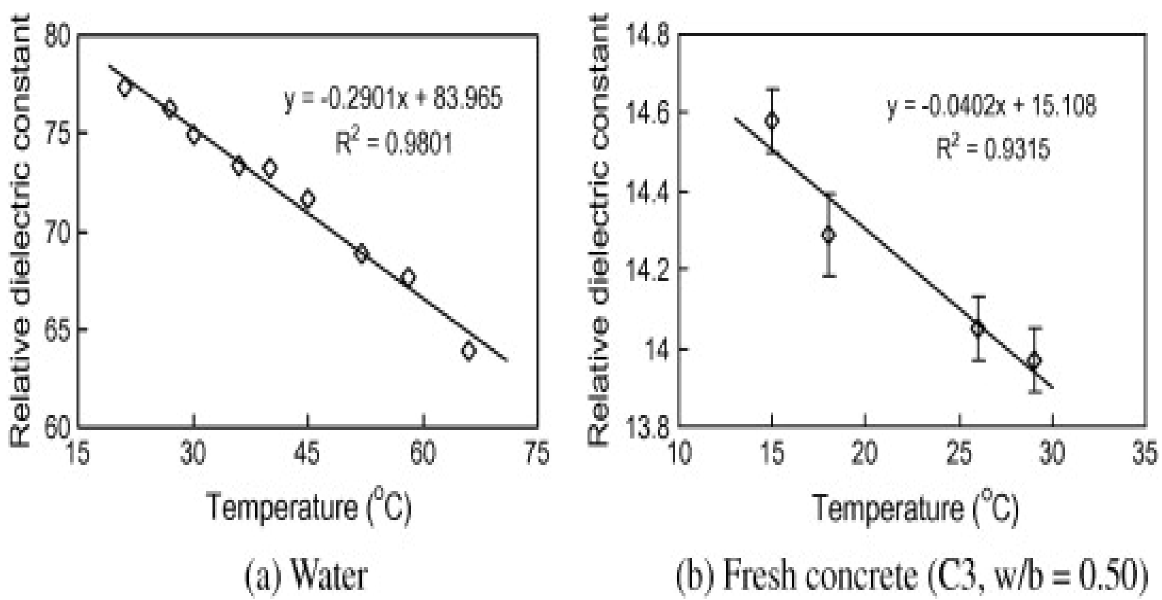


Figure 3 Relative dielectric constant of a) water b) fresh concrete at different temperatures

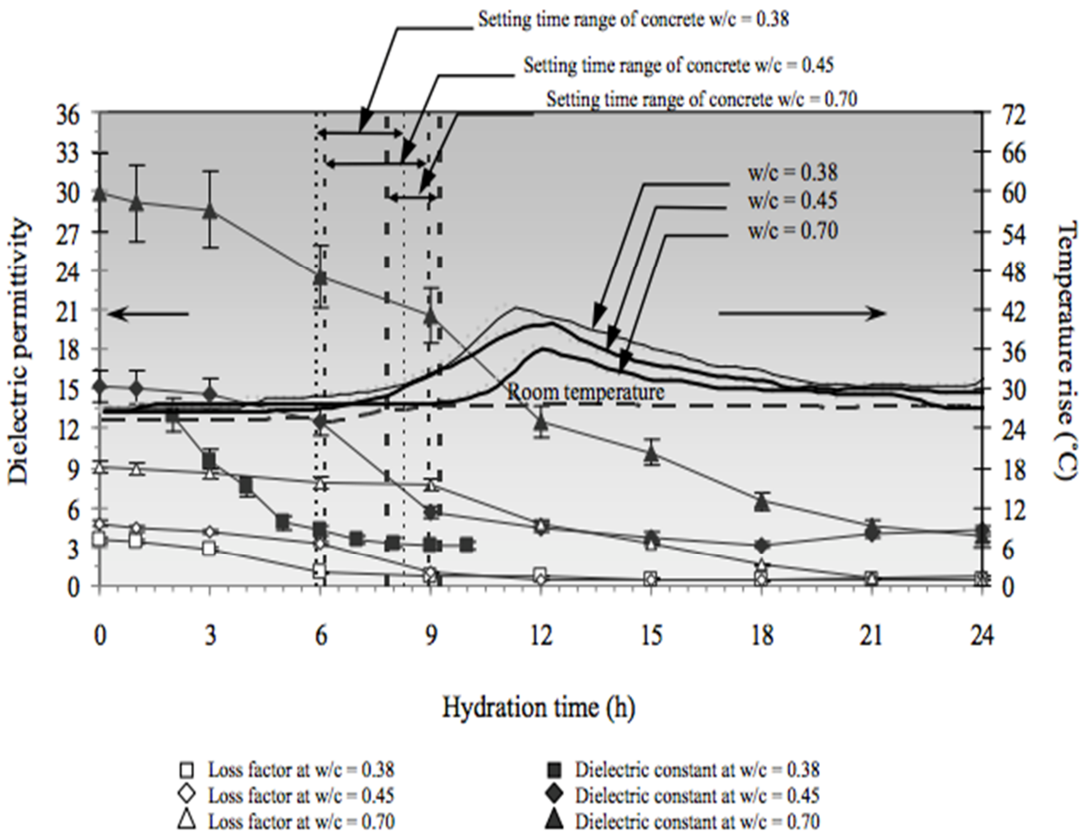


Figure 4 Dielectric permittivity of conrete's with different w/c over hydration time

CHAPTER 3

METHODOLOGY

3.1 Cementometer™ Properties

The Cementometer™ is stated to rely upon an imposed microwave frequency and the measured dielectric constant of the material to estimate the quantity of free water in cementitious material mixture (James Instruments 2010). The range of w/cm ratios the meter is capable of testing is reported to be from 0.35 to 0.65. The frequency cannot be adjusted within the Cementometer™ device and is not known. The Cementometer™ features a handheld console with a digital readout screen connected through a wire to two probes that are used to measure the dielectric of the mix. The internal software in the meter is hidden and cannot be accessed or modified.

The system is assumed to have one conductor and thus use a waveguide method to assess dielectric relative permittivity similar to a parallel plate capacitor. Although the company does not specify the design details, it is assumed that one probe of the device transmits the microwave frequency while the second probe receives the returned wave signal. This two-probe design creates a capacitor system similar to that shown in Figure 1 but with rods instead of plates. Figure 5 shows the Cementometer™ device, with its 5-inch-long probes spaced 1 inch apart. On the handheld display, the user can select the desired mode for measurement. An image of the device's display during different modes and stages in selecting the mode are shown in Figure 6. The four modes are described as follows:

1- **Direct Reading**: A unit-less reading used mainly to calibrate the meter. The maximum direct reading was found to be 1300, which is measured when the meter is in air. The direct reading changes based on the w/cm and mixture.
2- **User-Program**: This setting is used to calibrate the meter for different combinations of cementitious materials and solids. The meter can be calibrated for up to 9 different source material combinations, each calibration based on variation only in the water content of the mix. During each meter calibration for a given source material combination, a total of 45 direct reading measurements must be made.
3- **Type-I**: A precalibrated program from the manufacturer. The program is stated to be calibrated to a concrete mixture containing ASTM C150 (ASTM 2012a) Type-I cement as the sole type of cementitious material used in the mixture.
4- **Type-III**: A precalibrated program from the manufacturer. The program is stated to be calibrated to a concrete mixture where an ASTM C150 (ASTM 2012b) Type-III cement is assumed to be the sole cementitious material used in the mixture.

3.2 Meter Calibration

3.2.1 Source Material Used in the Study

One cement source, one fly ash source, four fine aggregate sources, and four coarse aggregate sources were investigated during this research study. All mixtures used an ASTM C150 (ASTM 2012a) Type II/V cement from Lafarge-Holcim's Devil's Slide Plant in Morgan, Utah. The fly ash (FA) is an ASTM C618 (ASTM 2012c) class F from Headwaters Navajo Plant. The cementitious material chemistries and particle size information can be found in Appendix A. The mixture proportions and specific ranges of w/cm used for calibrating the Cementometer™ are summarized in Table 2. The "Point Project" mixtures were re-created at the Utah Department of Transportation (UDOT) Central Materials Lab using the collected aggregates from the same plant sites for the actual reconstruction of I-15 interstate during the 2015-2016 year. Some of the aggregate sources changed during this time frame, so the date and location of the specific pit is shown in Table 2. The Cementometer™ was also calibrated and tested to two Harper Precast Concrete mix designs: CSCC100GZ which is a standard self-consolidating concrete (SCC) mixture and C2400FBGZ which is for Jersey barrier walls. "In-house" mixtures refer to those cast using the source materials at the University of Utah Concrete Laboratory. Aggregate properties can be found in Appendix A. All aggregates used for calibrating and testing were carefully prepared to Saturated Surface Dry (SSD) condition since NDT James Instruments Company recommends this in order to insure the most accurate output from the device. ASTM C127 (ASTM 2012a) was followed for conditioning the fine aggregates. Water used to batch concrete in batching plants was tap water, not deionized water, and thus the dielectric constant might differ slightly from the measured previously as was shown in Figure 2.

3.2.2 Concrete Calibration Mixing Procedure

During calibration, the Cementometer™ manual describes a step-by-step process for preparing the aggregates, batching aggregates based on their density, and adding increments of water to each desired w/cm ratio until the full range is met. Aggregates are expected to be added at SSD condition. Specifics of the manufacturer's calibration method are described in more detail in Appendix B. The Cementometer™ was calibrated for a total of the 14 different mixtures over the duration of this study, as listed in Table 2.

Due to the variable dielectric constant generated by different hydrating and absorbing materials in concrete, the manufacturer recommends calibrating the meter by using the same concrete mixtures with the actual aggregates and cementitious materials as the ones that will be used for future in-situ mixture measurements. This ensures that the meter's user-program generates the most accurate reading. For each combination of source materials to be calibrated in the user-program, the manufacturer recommends reading 9 w/cm ratios, starting from a 0.35 w/cm ratio, and then adding a fixed amount of water to produce increasing w/cm ratios up to 0.75. The manufacturer also recommends calibrating a batch size of 1 cubic foot of concrete. To generate the actual w/cm ratios during calibration, the manufacturer recommends that the aggregates start at SSD condition to avoid errors in the w/cm estimation due to user's estimated aggregates absorption. Also, during this calibration process, for each w/cm ratio reached in the mixture, the user must manually type in that w/cm value into the handheld device (shown in Figure 6c). After each w/cm value is entered in the system, the display then shows the direct reading value corresponding to the measurement (shown in Figure 6b). Because the direct reading value is expected to be highly dependent on slight alignment, orientation, location of the probe within the concrete, or even the distribution of solids between the probes, the manufacturer requires 5 different measurements per each w/cm value during the calibration. The steps found in Appendix B outline the procedure followed to prepare and mix the sample for calibration

3.2.3 Cement Paste and Mortar Calibration Curves

Among the mortar mixtures, a batch was made with air-dry sand instead of SSD condition sand during calibration. The values input in the software during the mixture with air-dry sand were not the actual w/cm values. Instead, the input w/cm value was based on the total water amount added. Figure 7 shows the direct reading obtained from the mortar and cement paste mixtures. The two mortar mixtures exhibited similar trends, with the direct reading rapidly decreasing at low w/cm and then tapering off at higher w/cm. The difference between the two plots is in further into the calibration process, the adjusted mortar direct reading seemed to increase and then decrease again, as compared to the decreasing trend in the SSD mortar. This difference in trend can be attributed to the time it takes the water molecules to seep through the pores in the sand.

In all three cases, the direct reading seemed to start at a higher value at low w/cm and decrease rapidly until intermediate w/cm, beyond which only a slight change in direct reading is observed with increasing w/cm. This could be attributed to the fact that at higher w/cm, the mixture segregated with most solids sinking to the bottom of the testing container. Hence, when calibrating mixtures at high w/cm, the meter is mainly reading a water mixture with a small content of other solids.

3.2.4 Concrete Calibration Curves

The 5 direct reading values per each of the 9 w/cm batched during calibration were recorded and plotted against the w/cm for the paste, mortar, and concrete mixtures. The results are shown in Figures 8 to 10. These figures demonstrate what the direct output reading values displayed during each calibration water addition step compared to the actual w/cm ratio for that calibration step.

3.2.5 Calibration Curves Regression Analysis

NDT James Instruments, Inc. states that the meter uses a straight line (linear) relationship to calibrate the actual w/cm mass ratio and the output of the device. To test the variability in the meter's calibration between the w/cm and direct reading, a regression equation of the actual w/cm mass values was plotted against the calibration direct reading values. The linear regression equations fit to the calibration direct reading values are shown in Table 3 along with each equation's R^2 values. The direct reading values obtained during the calibration demonstrate a poor linear fit, as can be justified by the low R^2 values for all the calibrated mixes. The R^2 values for the w/cm mass ratio prediction of the device were all less than 0.57 indicating there are either likely outliers or high variability in the calibration readings.

3.2.6 Discussion on Calibration Challenges

As an additional observation during calibration, there was a difficultly with inserting the probes in mixtures with w/cm ratios below 0.30 mainly due to the low workability (stiffness) of the mixture. Furthermore, when the w/cm ratios were higher than 0.55, all of the mixtures batched for calibration appeared to become segregated, with the coarse aggregates within the cement paste segregating and sinking to the bottom of the testing bucket. This can cause inaccurate direct readings since the volume of concrete between the probes no longer represents the mixture

The user also has the option during calibration to terminate the process at any w/cm before the full set of measurements was taken. In the case of an early termination, the device in the user-program mode was not able to display any values when the program was used to test concrete. Only one trial where the calibration was terminated before the 9 readings was done since calibrating the meter is a long and intensive process and another faulty program could not be risked.

Finally, a concern was noticed in the calibration process in that the Cementometer™ might record the same exact direct reading value for two different w/cm mixtures. For example, the direct reading recorded during calibration for w/cm ratios of 0.35 and 0.45 stored the same direct reading value. Thus, when later validating a mixture with a known w/cm of say 0.35, since the corresponding direct reading could be from 0.35 or 0.45 mixtures, the user-mode w/cm output sometimes jumped between 0.35 or 0.45.

3.3 Sensitivity of Direct Readings to Temperature and Mixing Time

Since the theory behind dielectric permittivity shows a dependence on temperature, it is hypothesized that temperature of the mixture during the testing may affect the value of the Cementometer™ output. Since the dielectric of concrete is also dependent on temperature, a simpler test of studying water alone with the meter against different temperatures from 1°C to 95°C was performed. In addition, both deionized water and tap water were both measured for this brief study. A linear correlation of increasing temperature to decreasing dielectric permittivity, similar to that found by (Shen et al. 2012), was expected. Rather than measuring pure dielectric values, the direct readings from the meter were recorded for the two water types. To obtain temperatures below room temperature, the water was chilled in a refrigerator prior to the reading. Then to obtain temperatures greater than room temperature, the water was placed in an oven set to 100 °C and measurements were recorded periodically as the water heated. With each Cementometer™ recording, the water temperatures were simultaneously measured using a Weber Instant-Read Thermometer probe with a +- 1 °C precision. Sample size and testing procedure were identical between the two water types and within each measurement. The results of the temperature versus direct reading of water measurements can be seen in Figure 11.

There was no trend on the direct reading values over the entire temperature range. There was also no specific trend on whether deionized water or tap water increases or decreases the direct reading relative to each other. The data visually appear to have a sudden drop at 30 °C, although logic behind this temperature drop value is not known. For values more common in an outdoor concrete placement environment, it appears there may be a linear negative correlation between the direct readings of the water to an increase in temperature. Due to the scatter of the points, this trend cannot be confirmed.

A time-dependency of the measurement was also hypothesized. During calibration, in order to make 45 different readings, the user takes on average about 2 hours to complete the measurements. During this time frame, the free water content and hydration phase of the mix may not necessarily reflect the proportions with which the mixture was just initially batched. Although the start time of calibration was not investigated in this report, another brief study was performed to see if the measured Cementometer™ readings would produce a different value over the course of mixing time with just investigating the direct reading values.

As mentioned in earlier sections, concrete electrochemical properties were found by other researchers to be dependent on the hydration phase and time (Makul 2013). Since the dielectric constant changes as the concrete hydrates, it is hypothesized that the meter may be sensitive to ionic concentration (related to conductivity) in the pore solution or the free water availability before being bound up into hydration products, as confirmed also by (Shen et al. 2016). The Cementometer™ is expected to be used just after mixing and prior to hardening stages of hydration, under conditions similar to the experiment conducted by Chen et al. and Shen et al.. A brief study was done to investigate the effect of longer mixing times (possibly due to longer transportation times or to remixing on-site) on the direct readings from the meter. It must be noted that mixing time is different than hydration time. With mixing, external energy is applied to shear apart particles and to aid in dissolution of the cement particles by exposing them to the hydrating water.

To test the time effect on the direct reading and other modes of the meter, mixtures of four different w/cm contents (0.35 and 0.40) were investigated after different mixing times. Each mixture was tested at 15 minute intervals up to 60 minutes. The results of both the direct reading values and mode I w/cm output values are shown in Figure 12. The direct reading values appear to slightly increase over time; however, the variability is still high in all measurements.

For further confirmation of the noticed variation and statistics, a one-way analysis of variance (ANOVA) was performed with a 95% level of confidence to analyze the difference (if it exists statistically) between the output values over the time interval. The analysis was performed only on the two w/cm contents (0.35 and 0.40) that had 3 replicate measurements at each time; the other w/cm contents tested only had 1 measurement at each mixing time. The results from ANOVA are most accurate when the variances of the samples compared are equal. The p-value displayed in Table 4 were calculated assuming an equal variance between the means of readings over time. This assumes that the variance of the readings of the two modes does not change over time. An unequal variance was assumed for the user-mode and direct reading values shown in Table 4. The choice of equal variances for the Type I and Type III modes and unequal variances for the direct mode and user-program becomes more clear in the discussion on the standard deviations of each mode and the scatter of the points. The high p-value shown in all modes indicates that either the variability is high or the output values at different mixing times up to 60 minutes cannot be distinguished based on this limited data set.

Table 2 Mixture Properties for Calibrating and Testing

Mix	FA Amount (% cementitious)	Moisture Condition of Sand	Moisture Condition of Coarse Aggregate	Aggregate Source*	Range of dosed w/cm ratios[¥]
In-House 'Paste 1'	0	-	-	Beck Street	0.35 to 0.75
In-House 'Paste 2'	0	-	-	Beck Street	0.30 to 0.39
In-House 'Mortar 1'	0	SSD	-	Beck Street	0.35 to 0.75
In-House 'Mortar 2'	0	Air Dry	-	Beck Street	0.25to 0.65
In-House 'Concrete 1'	0	SSD	SSD	Beck Street	0.30 to 0.65
In-House 'Concrete 2'	20	SSD	SSD	Beck Street	0.35 to 0.75
Harper SCC CSCC100GZ	30	SSD	SSD	Harper	0.35 to 0.75
Harper Barrier C2400FBGZ	30	SSD	SSD	Harper	0.35 to 0.75
The Point 6025E Oct 2015	25	SSD	SSD	Point East	0.35 to 0.75
The Point 6025W Oct 2015	25	SSD	SSD	Point West	0.35 to 0.75
The Point 6025E Nov 2015	25	SSD	SSD	Point East	0.29 to 0.57
The Point 6025W Nov 2015	25	SSD	SSD	Point West	0.35 to 0.60
The Point 7025E Apr 2016	25	SSD	SSD	Point East	0.35 to 0.75
The Point 7025W Apr 2016	25	SSD	SSD	Point West	0.35 to 0.75

*Aggregate properties can be found in Appendix A.
[¥] Dosed mass ratio does not include adjusted for aggregate moisture condition from what is stated in table.

Table 3 Calibration Linear Regression Equation and R^2 Values

Mix	Calibration Linear Regression Equation y= direct reading x=actual SSD w/cm mass ratio	R^2
In-House Concrete 1	y = -48.095x + 778.7	0.022
In-house Concrete 2	y = -682x +1189.5	0.480
Harper C2400FBGZ	y =-107.2x + 958.47	0.089
Harper CSCC100GZ	y =-230.13x + 984.58	0.325
The Point 6025E Oct	y = -123.53x + 839.97	0.043
The Point 6025W Oct	y = 48.4x + 717.6	0.015
The Point 7025E Nov	y = 143.26x + 713.9	0.031
The Point 7025W Nov	y = -159.56x + 838.1	0.530
The Point 7025E Apr	y = -922.53x +1196.9	0.571
The Point 7025W Apr	y = -444.93x +1041.4	0.550

Table 4 P-values from ANOVA Based on Influence of Mixing Time

w/cm	Direct Reading	User-program	Type I	Type III
0.35	0.979	0.286	0.208	0.681
0.40	0.594	0.924	0.334	0.344

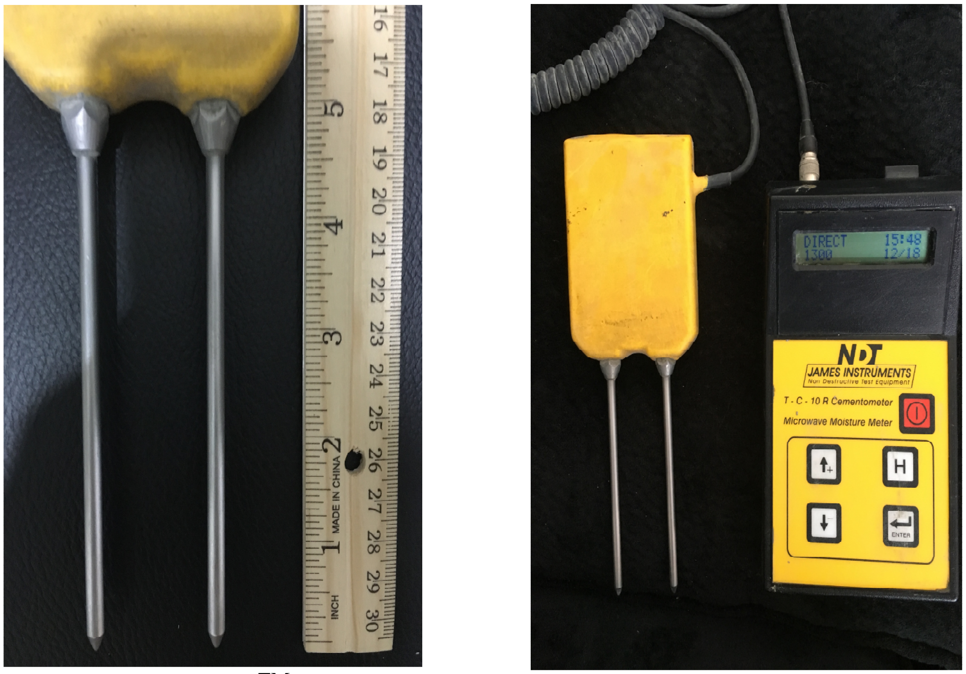


Figure 5 Cementometer^{TM} handheld meter and measuring probes.

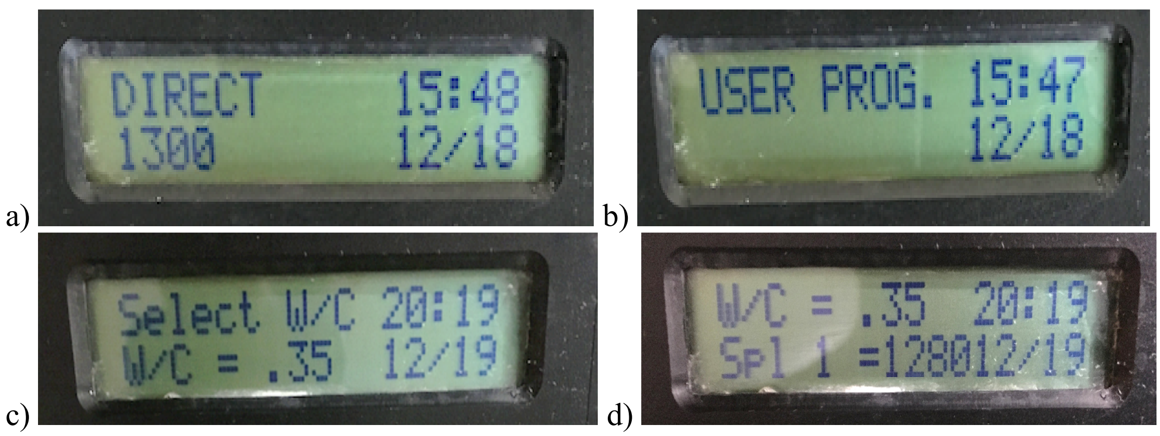


Figure 6 Cementometer Modes a) Direct (in air), b) User-Program c) Calibration reading w/cm value, and d) Device displaying the direct reading 1 out of 5 for that given w/cm value.

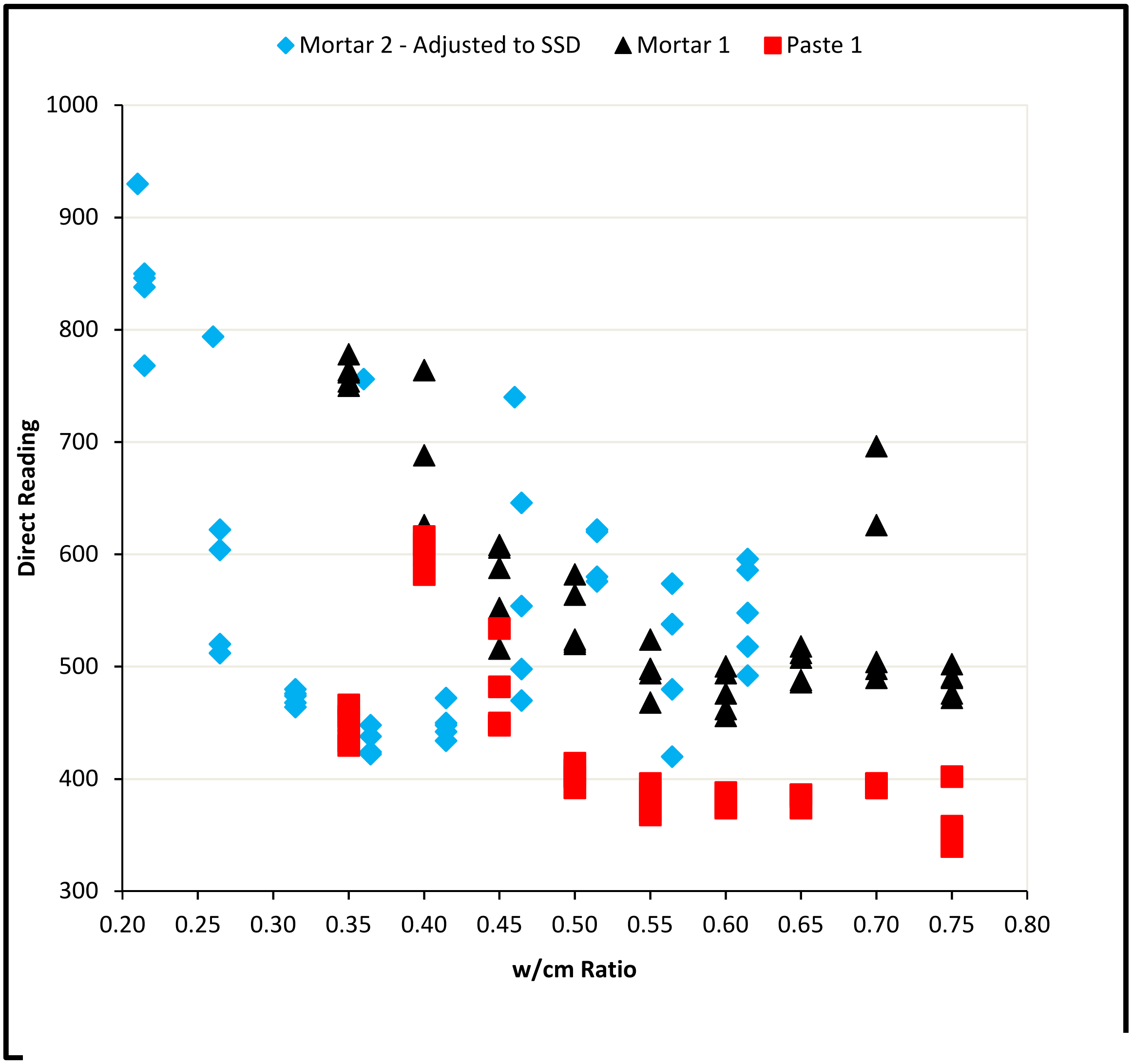


Figure 7 Direct readings during calibration of the mortar and paste mixtures plotted against total water-to-cementitious mass ratio.

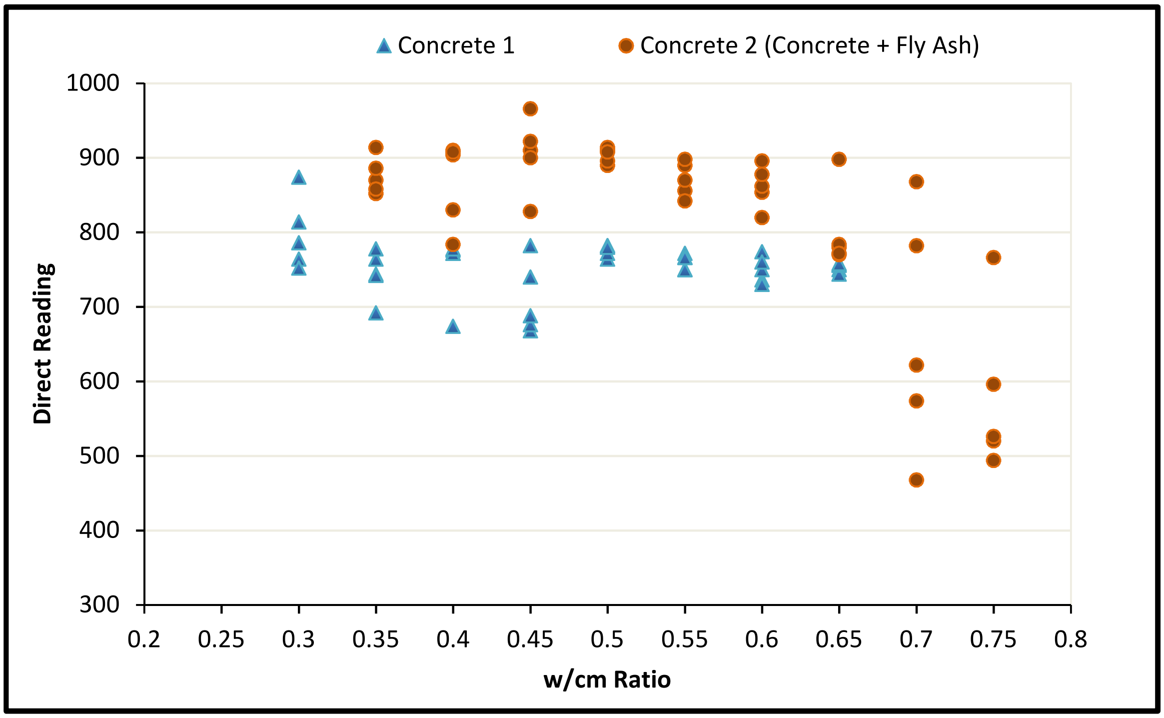


Figure 8 Direct readings during calibration of the Harper Precast mixtures plotted against total water-to-cementitious mass ratio.

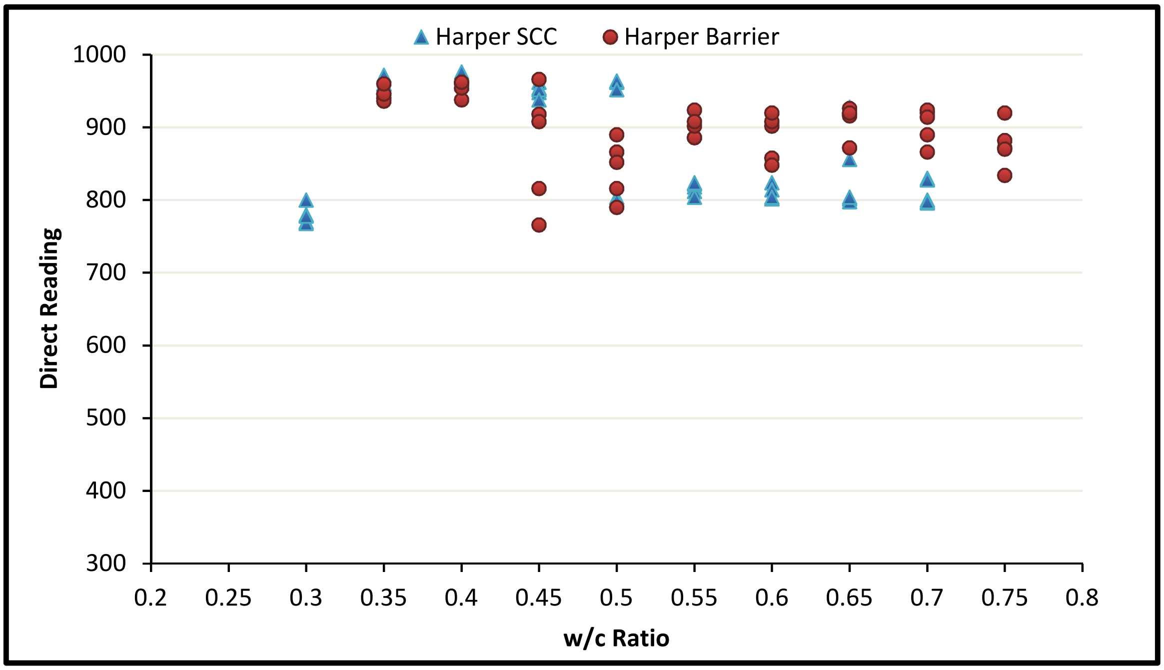


Figure 9 Direct readings during calibration of the Harper Precast mixtures plotted against total water-to-cementitious mass ratio.

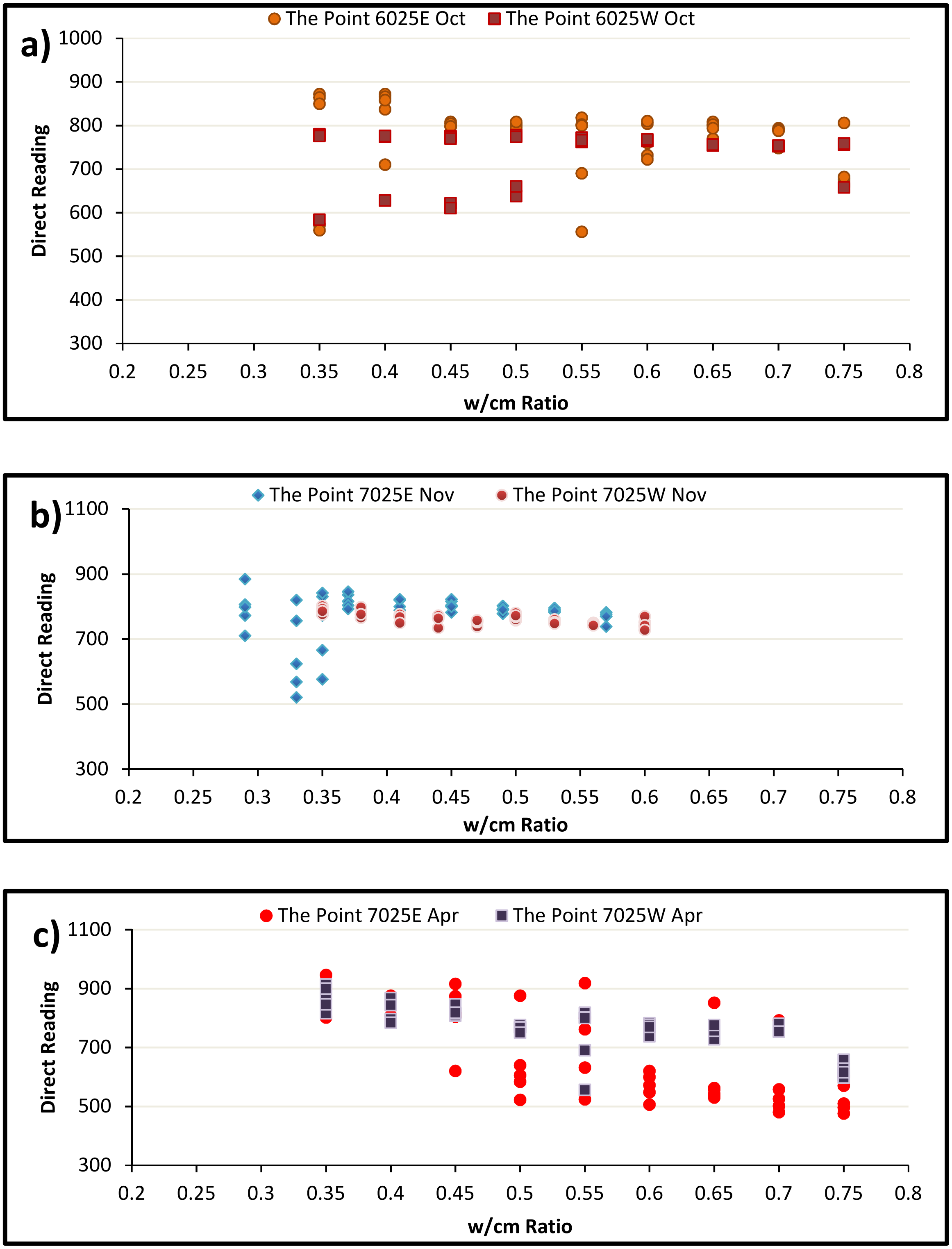


Figure 10 Direct readings during calibration of the Point mixtures plotted against total water-to-cementitious mass ratio.

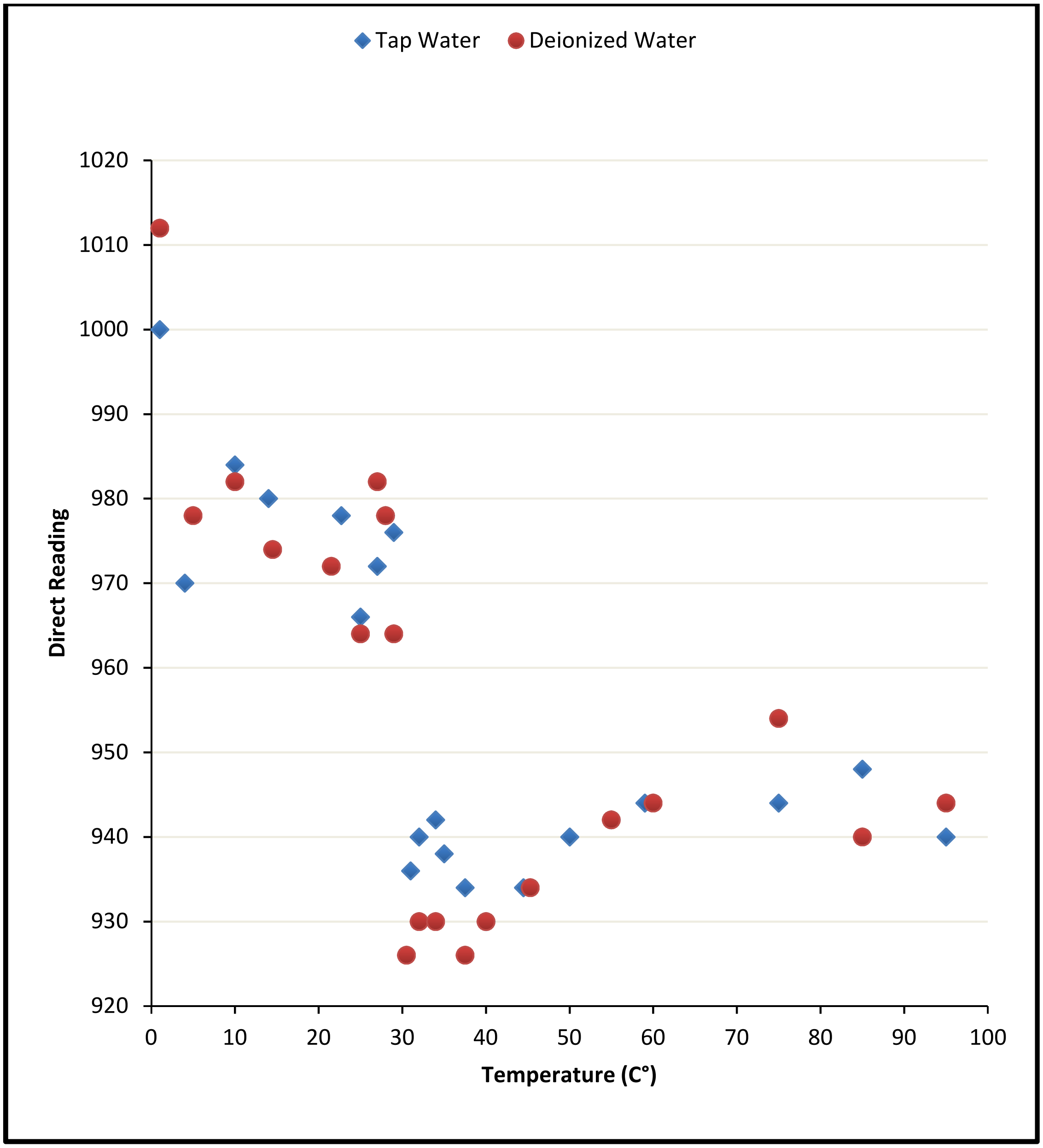
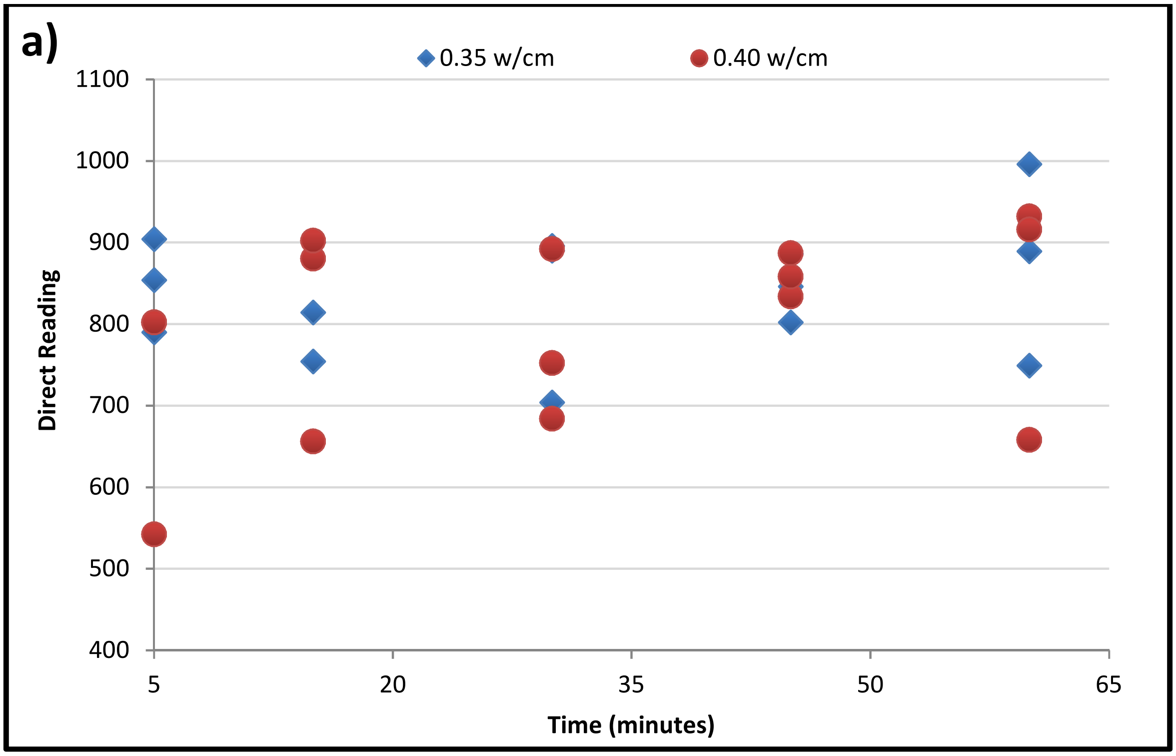


Figure 11 Meter direct reading on tap water and deionized water with varying temperature



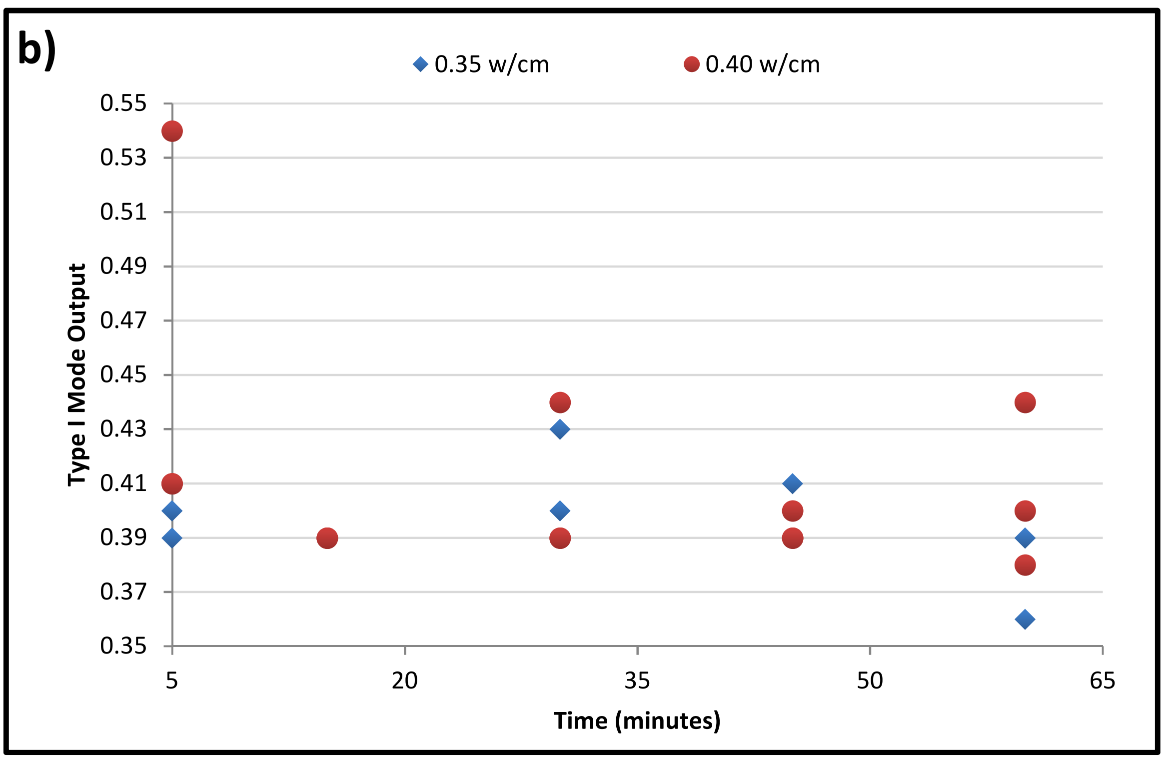


Figure 12 Meter reading a) Direct values and b) Type 1 mode predicted w/cm values.

CHAPTER 4

MEASUREMENTS WITH CEMENTOMETER ON SAND AND CONCRETE MIXTURES

4.1 General Measurements

The units of this direct reading are unknown, yet it was initially hypothesized that the direct reading was directly correlated to the dielectric constant. The direct output reading was found to be 1300 in air, while in tap water, the reading can range from 940 to 1000 depending on the temperature of the sample. Validation of the meter was performed to determine if the device can be used to distinguish between changes in the amount of free water in sand and concrete. A linear trend based on previously discussed literature was expected to be found between the output values and the water content in either a simple inert material, such as sand, or even a complex hydrating material, such as concrete. The output values from the Cementometer™ were recorded for sand and for concrete at varying water amounts. For sand, only the direct readings were recorded, while all four settings direct reading, calibrated user-program, mode I, and mode III) were recorded for the cementitious mixtures investigated

4.2 Measurements on Sand

Two types of sand (natural sand and a lightweight porous aggregate) were tested at varying water contents to verify if a trendline could be gathered between moisture content and the meter's direct reading output. The sands were dried for 24 hours in an oven at a temperature of 103°C and tap water at varying amounts was mixed at incremental levels to reach the varying moisture contents. The results of the direct reading outputs are shown in Figure 13a for the natural sand of 1.80% absorption capacity and 2.57 bulk specific gravity, and Figure 13b for the lightweight sand of 22.0% absorption capacity and 1.56 bulk specific gravity. A linear regression is shown in the figure in order to illustrate a possible correlation. This linear fit regression produced an R^2 value of 0.945 for the natural sand and 0.962 for the lightweight sand. Even if a linear regression is or is not the most fundamental fit, the high R^2 values indicate that the direct reading can be used with good precision to distinguish between amounts of free water content in sand. In that absorption capacity change, we can see that it has the nonlinear function and its not changing significantly, similar to the results of section 2.4.

4.3 Measurements on Concrete

Once calibrated, various cement, mortar, or concrete mixtures listed in Table 2 were rebatched at specific w/cm values to verify if the Cementometer™ would predict the same w/cm content as was actually batched. A total of 195 mixtures were tested for this validation, of which 157 are concrete mixtures with w/cm ratios varying from 0.30 to 0.55. The number of measurements per w/cm is presented in Figure 14. The same material sources and mix designs were used as the calibrated mixtures shown from Table

2. The actual w/cm was calculated based on batched weights created in the lab or retrieved from the batch plant before any additional water was added and based on the SSD condition of aggregate. Mixtures for validation were mixed and measured in the same location and with similar room temperatures as that mixture was calibrated in.

Once the mixtures were mixed, a representative sample was placed in a plastic bucket and the meter's probes were inserted in the mixture. The four possible mode outputs were then recorded. The bucket size used for validation was also identical to the same bucket used when calibrating that mixture to ensure consistency.

4.4 Measurement Results

The user-program, mode I, and mode III outputs from the device displayed anticipated water-to-cement contents; as such, these mode outputs were plotted against actual w/cm content. The validation measurements for all concrete mixtures are plotted in Figure 15. A value of "0" shown as the output y-axis in these figures was actually "out of range" (OOR) displayed on the device. When calculating statistics on the outputs, all of the OOR values were omitted, so some of the sample sizes were smaller than actually measured. Of the 157 concrete mixtures tested, the user-program mode read OOR 34% of the readings. The Type I mode only read OOR 1 time and Type III never read OOR.

Although the Cementometer™ appeared to be correlated to the direct reading of sand, visibly it can be seen from Figure 15 that the Cementometer™ rarely predicted the actual w/cm when used in cementitious mixtures. From the figure, the user-mode output visually appeared to have the highest deviation from the actual w/cm, followed by the Type I mode and then Type III. Visually, Type I mode appears to over predict when the actual w/cm is below 0.40 and under predict when the actual w/cm is above 0.40. Type III mode appears to over predict across most of the range of w/cm in Figure 14 tested. Several statistics were calculated to determine the magnitudes of precision and accuracy for these modes in distinguishing between and predicting different w/cm mixtures.

4.4.1 Regression Analysis

A linear regression was fit to the three different w/cm output modes of the Cementometer™. The R^2 value is calculated in this case for the linear fit to determine if there is a good linearity correlation between the actual w/cm and the output w/cm value from the meter. Unlike the regression equations shown for calibration, these only include w/cm output values based on the validation measurements. The linear fit is not shown graphically on each plot, but does correspond to the data shown in Figure 15. As summarized in Table 5, all three modes had a near zero R^2 value, indicating that there was little to no linear correlation between each of the Cementometer™ modes with respect to the actual w/cm. Table 5 also summarizes the mean and standard deviation values of the entire concrete validation data set.

The standard deviation in Table 5 represents the magnitude of variation in w/cm value for 68% probability, assuming the meter mode is normally distributed about its average w/cm output. The magnitude of standard deviation provides some indication as to how much variation a user can expect in the output display for any given mixture. The standard deviation was 0.02 for both the Type I and Type III modes and a much higher standard deviation of 0.12 for the user-program mode, despite the user-program mode being pre-calibrated to the exact same mixture components. It reflects the high variability that the user-program has in differentiating a unique w/cm value of a given mixture.

4.4.2 Relative Dielectric Constant Calculations on Sand and Concrete

Since it is theorized that the meter's direct reading is related to the dielectric constant of the mixture, the calculated dielectric constant using Equation (3) was plotted against the direct reading for the sand-only mixture and concrete mixtures, shown in Figure 16 and Figure 17, respectively. In each case, the mixture was modeled as a three-phase material that consisted of solid, liquid, and gas. V_1, V_2, and V_3 are calculated from the mixture proportions and ε_1, ε_2, and ε_3 are the dielectric constant of the liquid, solids, and gas with values obtained by Chen et al. as 78.16, 6.00, and 1.00, respectively. In the sand mixture test, the measured volume of air was approximately 35% of the total container volume. As water was added to the oven-dry sand, the volume of air was reduced and replaced by the volume of water added. Figure 16 shows the calculated dielectric constant from Equation (3) and the volumetric ratio of water-to-solids (sand-only) plotted against the direct reading from the Cementometer™. According to Wang et al., the dielectric constant of soil with moisture content below the transition moisture content should be modeled using a different relationship. For that reason, calculations of dielectric parameters prior to the sand reaching an SSD state were ignored and only the trend after the SSD state was studied. A linear approximation was suggested at this time, although other correlation regression equations may explain the data measurements more accurately. Even for a linear approximation, the regression's R^2 value indicates a strong relationship is found between the direct reading and calculated relative dielectric constant for sand. Similarly, the direct readings were plotted against the calculated dielectric constant from Equation (3) for concrete. These are shown in Figure 17 a and b. The equation as expected predicts a linear regression with a good correlation to the water content in the concrete. However, unlike the direct readings in sand and water, the direct readings from concrete do no indicate any correlation with water content or with the dielectric prediction equation.

Table 5 Actual to Predicted W/CM Ratio Statistics of Each Meter Mode

Mode	Linear Regression $\mathbf{R^2}$	Mean μ	Standard Deviation σ	Sum of Square Error SSE
Actual	-	0.41	0.04	-
User-Program	0.003	0.54	0.12	3.60
Type I	0.065	0.40	0.02	0.323
Type III	0.029	0.47	0.02	0.897

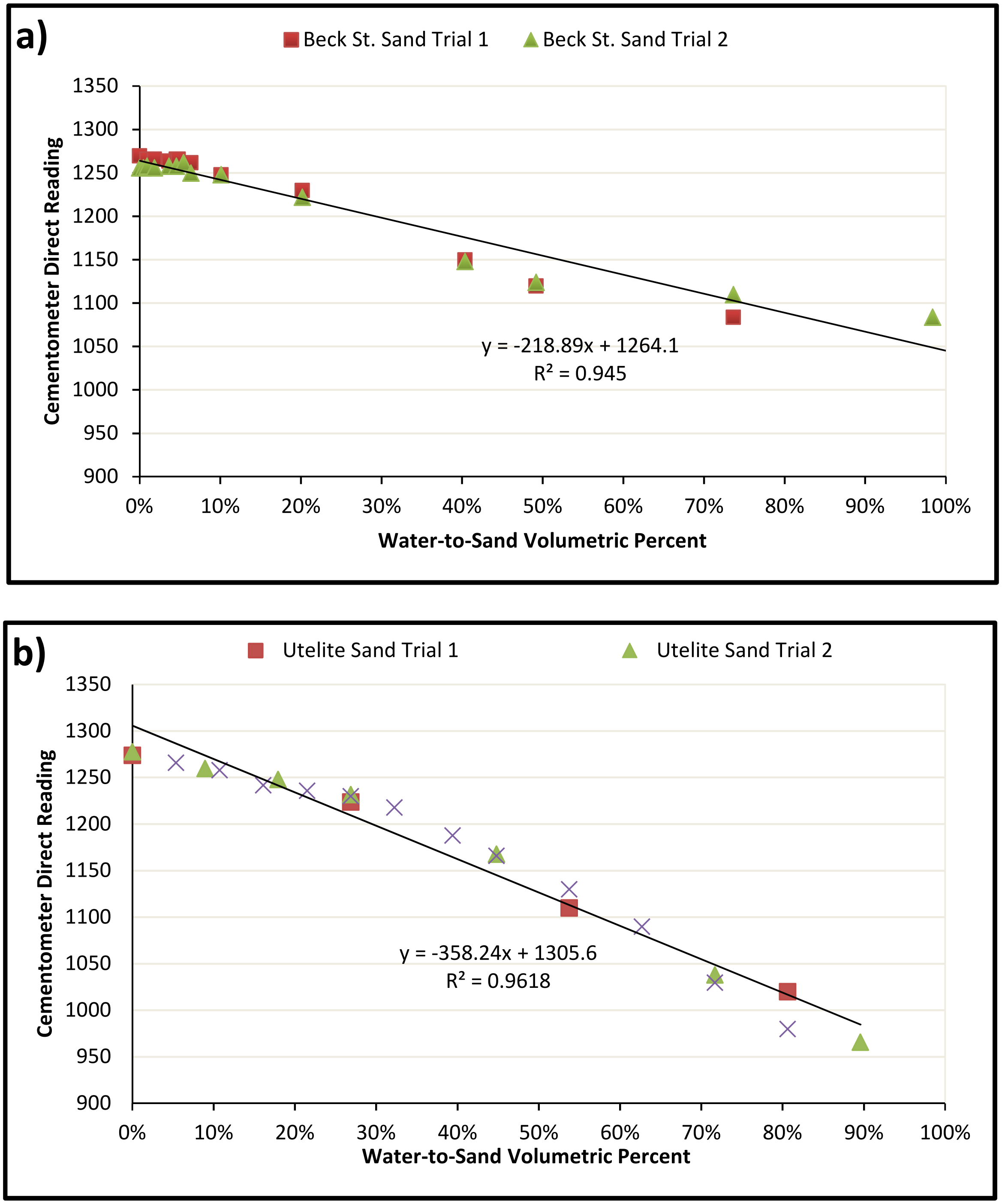


Figure 13 Sand used in experiment a) Beck St. natural sand b) Utelite lightweight sand.

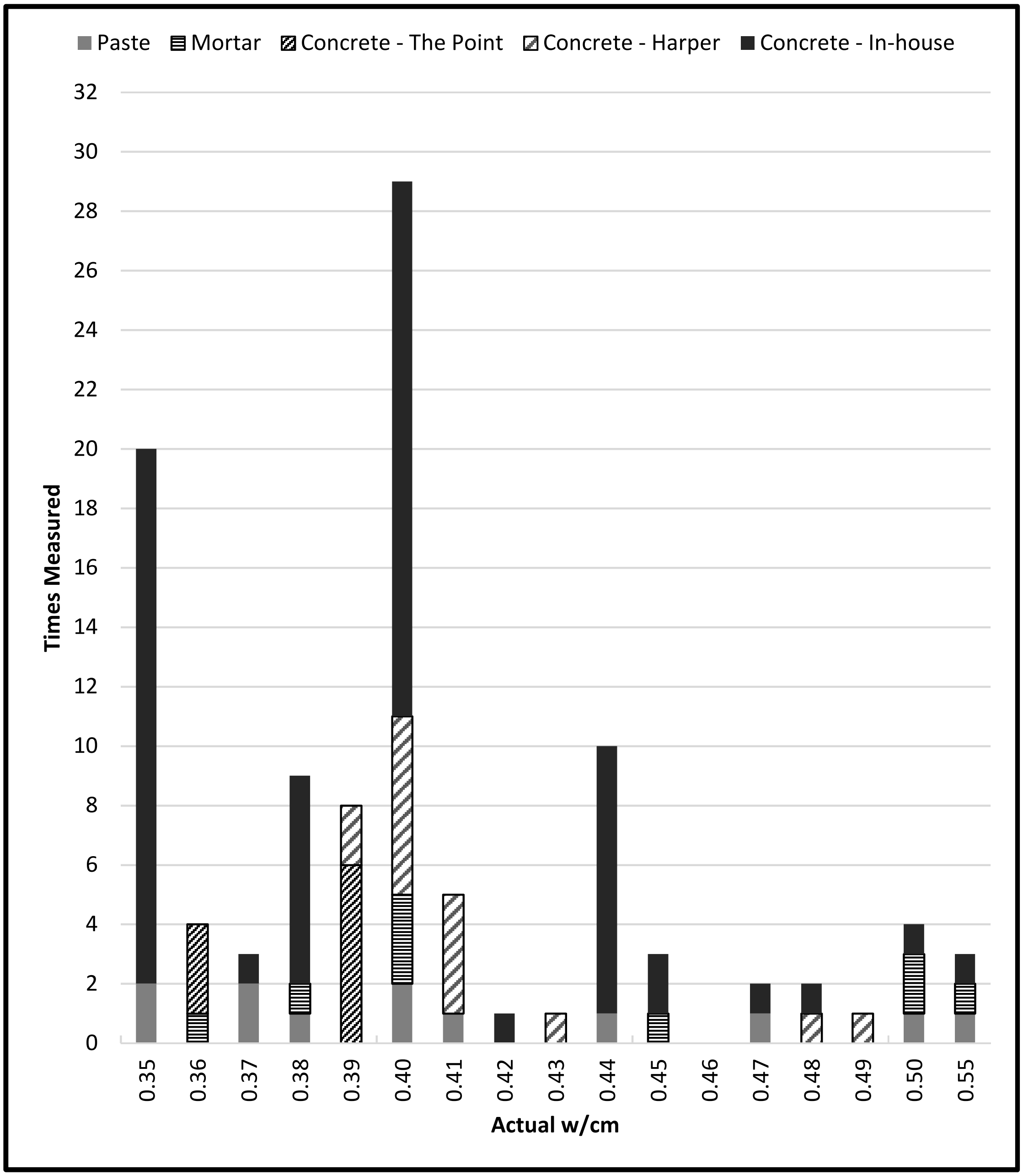


Figure 14 Measurement frequency of all cement mixtures tested for validation of the Cementometer^{TM}.

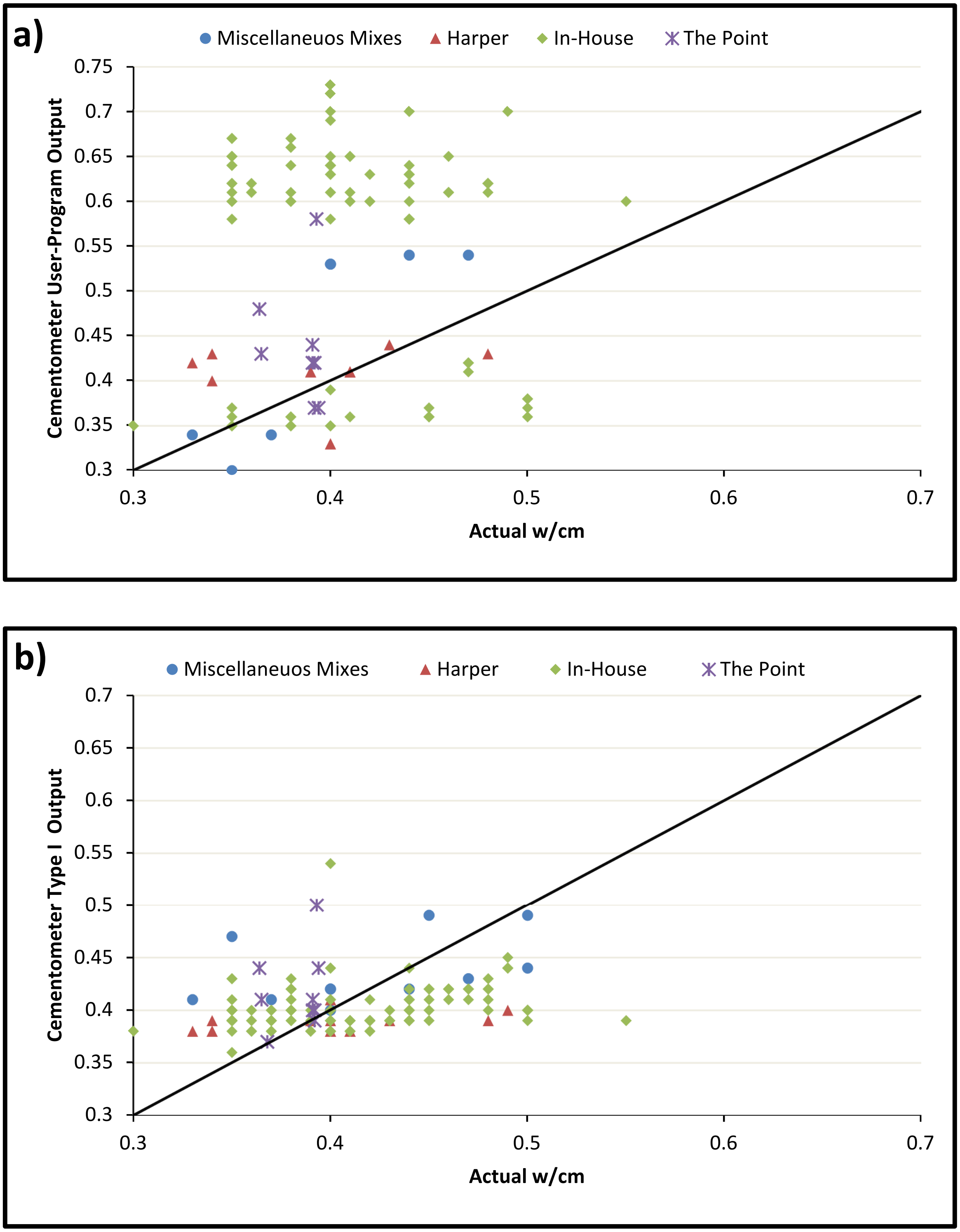


Figure 15 Measurement results a) User-Program b) mode I and c) mode III.

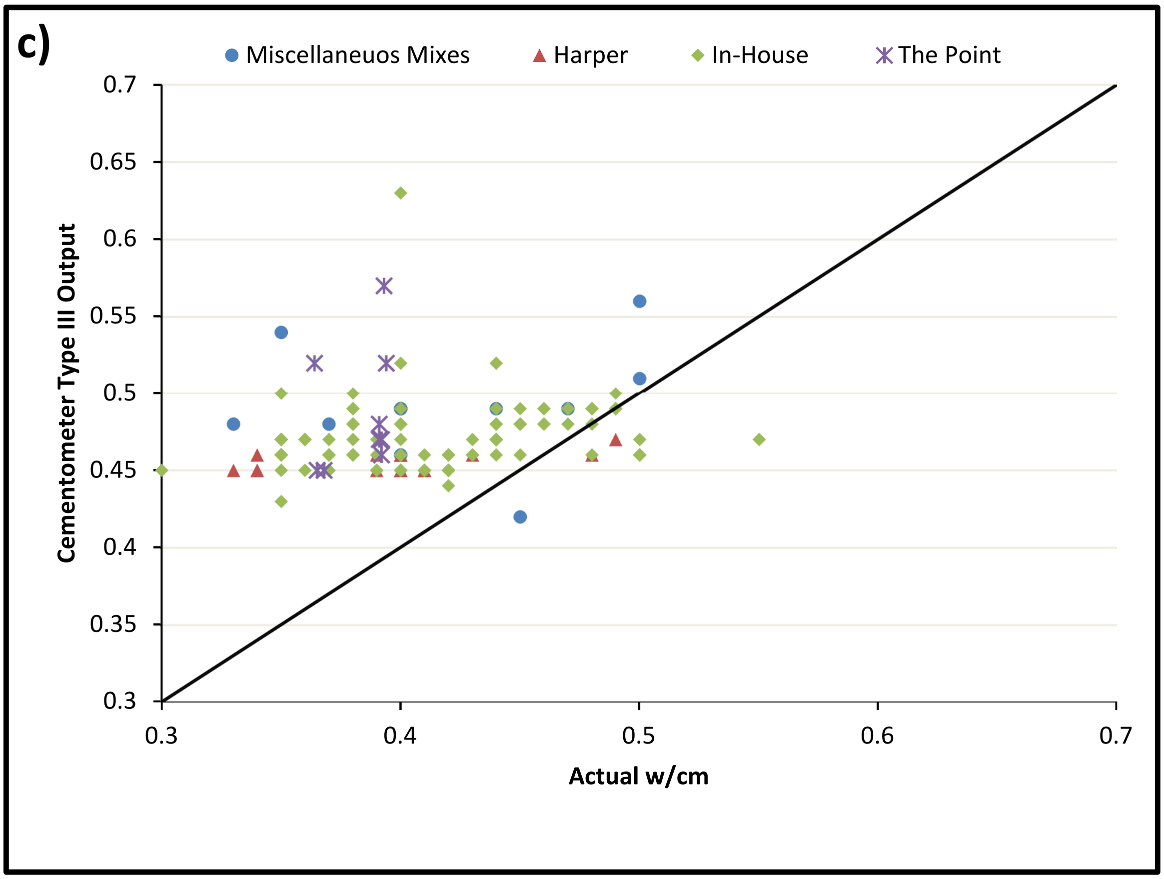


Figure 15 Continued

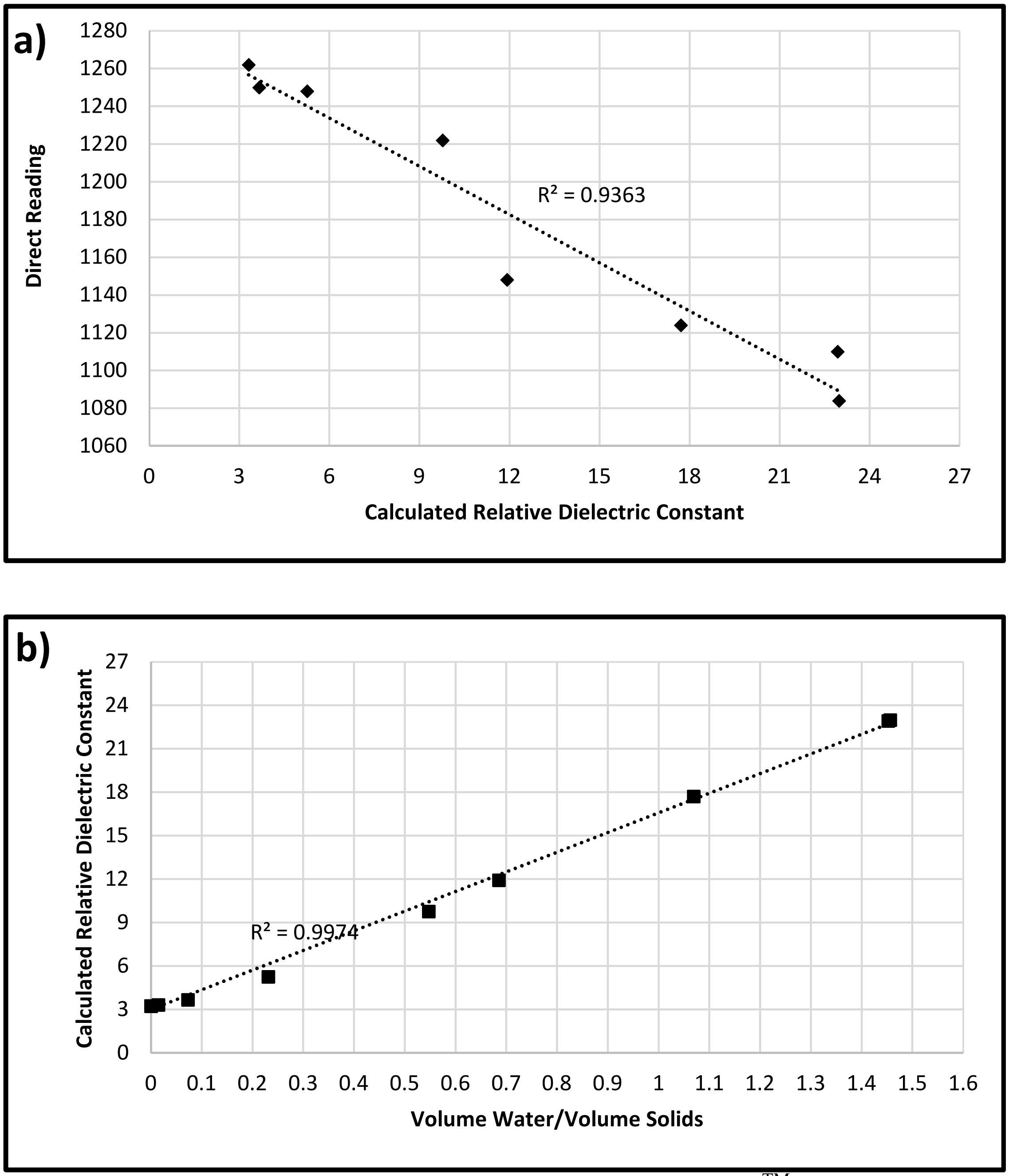
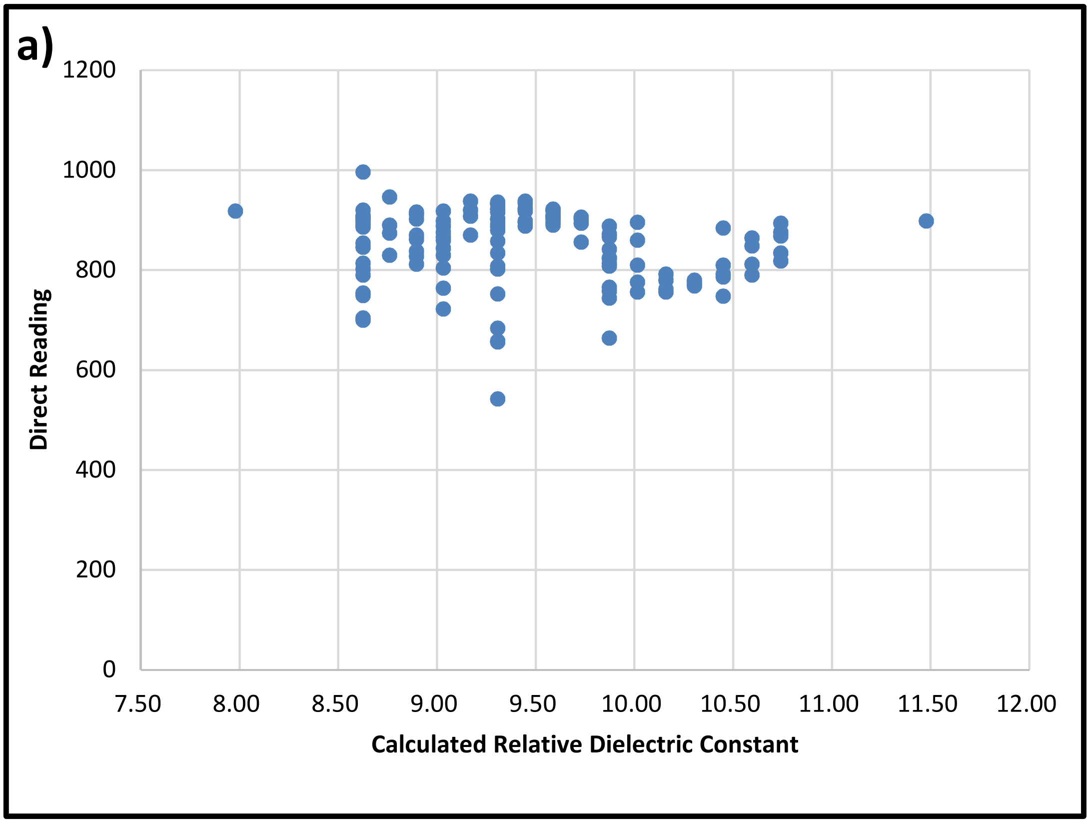


Figure 16 Sand measurements a) Direct reading from Cementometer^{TM} and b) Volumetric ratio of water to solids versus the calculated dielectric constant.



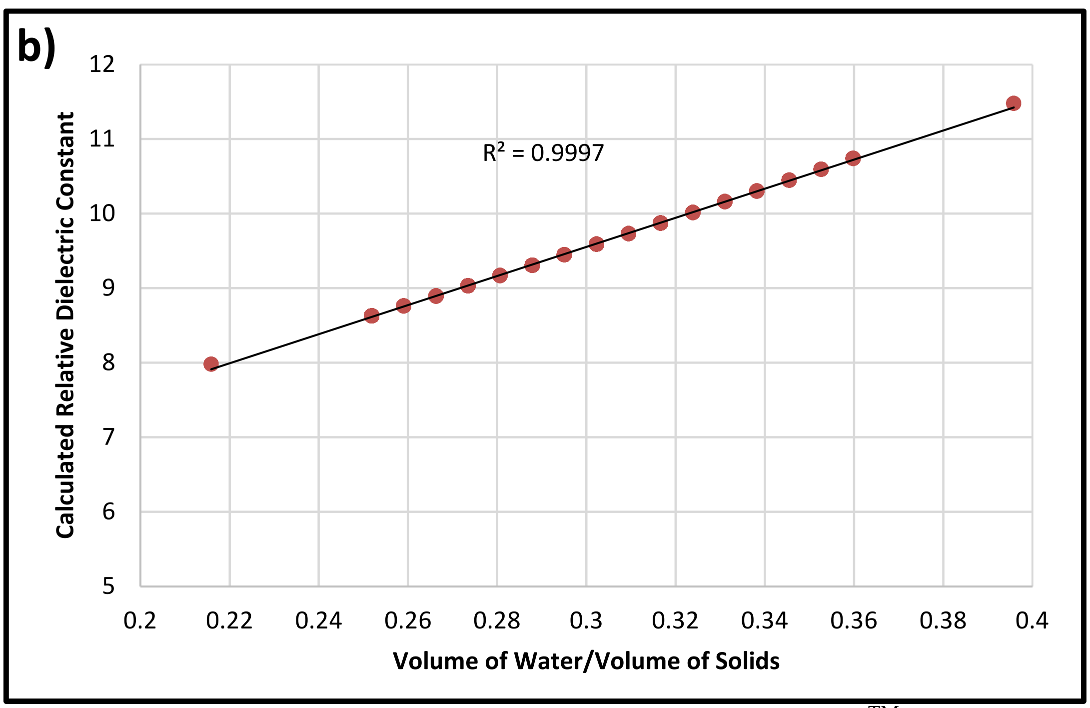


Figure 17 Solids measurements a) Direct reading from Cementometer^{TM} and b) As volumetric ratio of water to solids versus the calculated dielectric constant.

CHAPTER 5

ADDITIONAL STATISTICAL VERIFICATION

5.1 Statistical Analyses

Although it was noted by visually investigating the results in the previous chapter that the mode output of the Cementometer^{TM} does not appear to be correlated to the actual w/cm content, additional statistical analyses were performed to verify the precision and accuracy of the Cementometer^{TM}. Six different analyses were done on all concrete mixture validation results, excluding any OOR readings. A regression analysis, standard deviation, and absolute difference were used to assess the precision of the Cementometer™ modes in order to determine whether the meter can differentiate between two similar water contents. Then a t-test analysis along with the sum of squared error (SSE) were used to assess the accuracy of the three different modes in terms of whether the Cementometer™ might present a w/cm value close to the actual w/cm. Finally, a confidence interval was calculated based on the t-test, which helps to present statistically the range of expected w/cm values that the Cementometer™ may give as an output for a given mixture's actual w/cm ratio.

5.1.1 Absolute Difference and Sum of Square Error (SSE)

The absolute and average differences between the actual w/cm and the output w/cm of the three modes were calculated and are plotted in Figure 17. The SSE represents the squared difference of the error of the individual output w/cm values from the actual w/cm. The SSE values for the different w/cm measured are shown in Figure 18. The difference between the absolute difference and the SSE calculations is that the SSE sums up each individual measurement's error magnitude rather than just direction. Summing the squared error values gives a better representation of the total magnitude of variability. The absolute difference does not indicate whether the meter over or under predicts. Instead, both the absolute difference and the SSE do give an idea of the precision of the device relative to the actual w/cm content. It must be noted that some measurements were excluded from the analysis because they read OOR, and as such, some user-program measurements appeared to have a low average difference and low SSE from the reduced sample size. An example calculation of the absolute difference and SSE can be found in Appendix D.

The user-program had the highest differences, particularly the highest average difference of 0.23 and highest SSE at an actual w/cm of 0.35. The user-program had the highest difference and SSE across most w/cm mixtures when compared to Type I and Type III mode outputs. The smallest average difference of 0.01 w/cm content occurred for the Type III mode at w/cm mixtures of 0.48 and 0.49. The next smallest average difference of 0.02 occurred with the Type I mode at w/cm mixtures of 0.37, 0.39, and 0.40. The Type I and Type III modes had SSE values below 0.05 for most w/cm mixtures. The low absolute difference and SSE values from the analysis may suggest that the meter's Type I and Type III may be preferred due to the lower variability and higher precision. However, the previously identified R^2 value indicated linear correlation cannot be used to differentiate small changes in w/cm content.

5.1.2 T-test for Each w/cm Ratio

A one-sample t-test is a statistical procedure used to compare the mean of a set of data to a specific value, such as the actual w/cm ratio for that mixture. The null hypothesis is H_0: $x = x_i$ where x is the actual w/cm ratio being studied and x_i is the mean of the meter mode's output sample values. The mean meter output of each w/cm tested in Figure 13 was compared to the actual w/cm of that mixture. For example, the mean of the Type I output from the meter for all concrete mixtures batched with a 0.35 w/cm was calculated to be 0.39 for a sample of 19 mixtures. An example calculation is shown in Appendix D.2. A p-value can be used to determine the probability for when the Type I mode output for those mixtures is equal to 0.35. This hypothesis can be either rejected or not rejected, based on whether the calculated p-value is less than or greater than a specified level of significance. Table 6 shows the calculated p-values for each batched w/cm and each testing mode. Only the modes which produce w/cm values (i.e., User-program, Type I, and Type III) were used in this t-test. The level of significance in this study was selected to be 0.05, meaning that the null can be either rejected or not rejected with 95% confidence. Thus, all scenarios where the p-value was less than 0.05 means the null is rejected, or that statistically the output w/cm value is not equal to the batched w/cm . As can be seen from Table 6, the p-values for most of the batched w/cm mixtures were all low, indicating that the mode could not predict the actual w/cm. While some of the p-values were higher than 0.05 indicating that the hypothesis cannot be rejected, in some of these cases the sample size was low and/or the variability among the sample set was high. These high p-values may be false positives, or called “type-1 errors” with the t-test statistic calculation.

5.1.3 Confidence Interval

The confidence interval was calculated to represent the predicted range of w/cm values that the Cementometer™ gives with a 95% confidence. This is calculated based on the meter mode’s average output w/cm value plus or minus the calculated w/cm difference associated with a 95% t-distribution for that batched w/cm ratio data set. The specific equation used for calculation for the confidence interval is shown in Appendix D.3.2. The confidence interval plots for each mode are shown in Figure 19. Among the three modes, the confidence interval range for the user-program has a wide range of up to a 0.388 difference in w/cm values occurring for mixtures such as at 0.36 actual w/cm content. Type I and III both visibly demonstrate a narrower range of possible w/cm values for any given mixture. The confidence interval range width for any given w/cm mixture measured with the Type I mode can vary across 0.08 w/cm values and with a Type III varies up to 0.07 w/cm values.

Very few of the measurements showed a range that lies across the actual w/cm of the mixture. With Type I, the only w/cm ratios that overlap the actual w/cm content with 95% confidence are 0.39, 0.40, and 0.45. With Type III, the ratios that overlap with actual w/cm contents are 0.45, 0.48, 0.49, and 0.50. Those w/cm contents which overlap the actual w/cm are also the only ones which had a p-value equal to or greater than 0.05.

Although the confidence interval and p-value analysis may suggest that the meter can be used at these specific batched w/cm mixtures, the risk is still high when using the meter's modes at these w/cm values.

5.1.4 T-test for Entire Mode

A two-sample t-test is a statistical procedure used to compare the mean of a set of data to the mean of another set. The null hypothesis here is: $H_0: \mu = \mu_0$ where μ is the population mean of the actual w/cm ratios and μ_0 is the mean of the entire mode's output population values. The mean meter output of all the w/cm tested in Figure 13 for each mode was compared to the mean of all actual w/cm of the mixture. For example, the mean of all the Type I outputs from the meter for all concrete mixtures tested was calculated to be 0.404, while the mean of actual batched w/cm ratios for the same 157 mixtures was 0.412 (the equation and example of the t-test calculation can be found in Appendix D). This sample t-test can be used to roughly estimate if an output w/cm value from a mode such as Type I will be a similar value to the actual w/cm value that might have been batched. The results of this t-test for comparing the entire mode to the actual batched w/cm values are summarized in Table 7. Since the p-values are again significantly low for all three modes (less than 0.05), we can reject the null and conclude that statistically, the output for these modes is not equal to the actual w/cm ratios.

5.2 Discussion on Statistical Findings

All three modes demonstrated high variability and low accuracy. The Type I mode appears to produce w/cm values on average around 0.40, and with a 95%

confidence that the Type I mode may display readings between 0.37 and 0.46 regardless of the mixture that was created. Since this range of output w/cm values is typically seen in most mixtures in the field, it is likely that one can assume the device is accurate. However, due to the low R2 values and low p-values, it is proven that all of the modes: the Type I, Type III, and User-Program modes, cannot be used to distinguish between mixtures of similar w/cm content and were in fact not accurate nor precise at predicting or differentiating between unique actual w/cm values.

5.3 AASHTO T 318-02 Concrete Microwave Test

One of the common methods currently used by the industry for quality assurance of in-situ water content is the AASHTO T 318-02 microwave test. While the Cementometer™ does produce a reading within a few seconds and is displayed as a w/cm value, the AASHTO test requires approximately 15 minutes to complete the measurement and similarly relies on microwave technology in determining moisture content.

The AASHTO test was performed as an additional study to simultaneously compare with the Cementometer™ recorded mode output values in the prediction of actual w/cm contents. A smaller sample set of five separate w/cm mixtures were batched to be separately analyzed using the AASHTO test. The output from the AASHTO standard actually gives only moisture content as a percent, but a “calculated w/cm ratio” of these five mixtures was also made based on the mixture proportions, the measured unit weight of the concrete, and specific gravities of material components. An example of the calculated w/cm ratio based on the AASHTO test result is described in Appendix C.

Figure 20 shows the moisture content values of the AASHTO microwave method along the actual and calculated w/cm of each mixture. Although this study had a limited number of data points, a linear trendline was fit through the actual w/cm contents for this AASHTO method's moisture content to provide a rough estimate on the correlation or trend that might occur between the moisture content and actual w/cm content. This trendline had a R^2 of 0.62 which is significantly higher than any linear trendline correlation from the Cementometer™ mode outputs versus actual w/cm ratio. Furthermore, a linear regression between the moisture content of the AASHTO method and calculated w/cm was a better correlation (R^2 of 0.99) than the correlation between calculated w/cm and actual w/cm ratio. To verify the accuracy of the results of this AASHTO microwave test, a t-test and SSE analysis were also calculated between the actual w/cm and calculated w/cm. The hypothesis tested was that the mean difference of the actual w/cm and calculated w/cm is equal to zero. As can be seen in Table 8, the p-value obtained for this AASHTO method is much higher than what was calculated for each of the Cementometer™ modes. With such a small data set, the p-value statistic may not be strong enough to conclude from. Yet, due to the higher R^2 value, low SSE, and the failure to reject the null hypothesis, this AASHTO method is preferred over the Cementometer™ device as a more accurate or more precise method to predict actual w/cm content of a mixture.

Table 6 P-values for the Validation Measurement w/cm Ratios

w/cm	User-Program	Type I	Type III
0.35	0.000	0.000	0.000
0.36	0.043	0.027	0.001
0.37	*	0.000	0.000
0.38	0.013	0.000	0.000
0.39	0.154	0.132	0.000
0.40	0.000	0.727	0.000
0.41	0.054	0.000	0.000
0.42	0.003	0.000	0.000
0.43	*	0.001	0.001
0.44	0.000	0.000	0.000
0.45	0.001	0.108	0.140
0.46	0.007	0.000	0.003
0.47	0.318	0.000	0.001
0.48	0.019	0.000	0.793
0.49	*	0.003	0.704
0.50	0.000	0.004	0.285

* One or less data points available

Table 7 T-test Parameters for Entire Mode Data Set of Concrete Mixtures

Mode	Sample size	Mean Difference	Standard Error	T-value	p-value	Hypothesis $X_{mode} = X_{Actual}$
User-Mode	108	0.123	0.086	11.47	0.000	Reject
Type I	156	-0.008	0.036	2.02	0.044	Reject
Type III	157	0.059	0.036	14.80	0.000	Reject

Table 8 Statistic for AASHTO Microwave Test to Actual W/CM

Data	Sample Size	Mean Difference	Standard Deviation	T-value	p-value	Hypothesis $\mu_{calculated} = \mu_{actual}$
Microwave Test	5	0.0025	0.0378	0.122	0.909	Do not reject

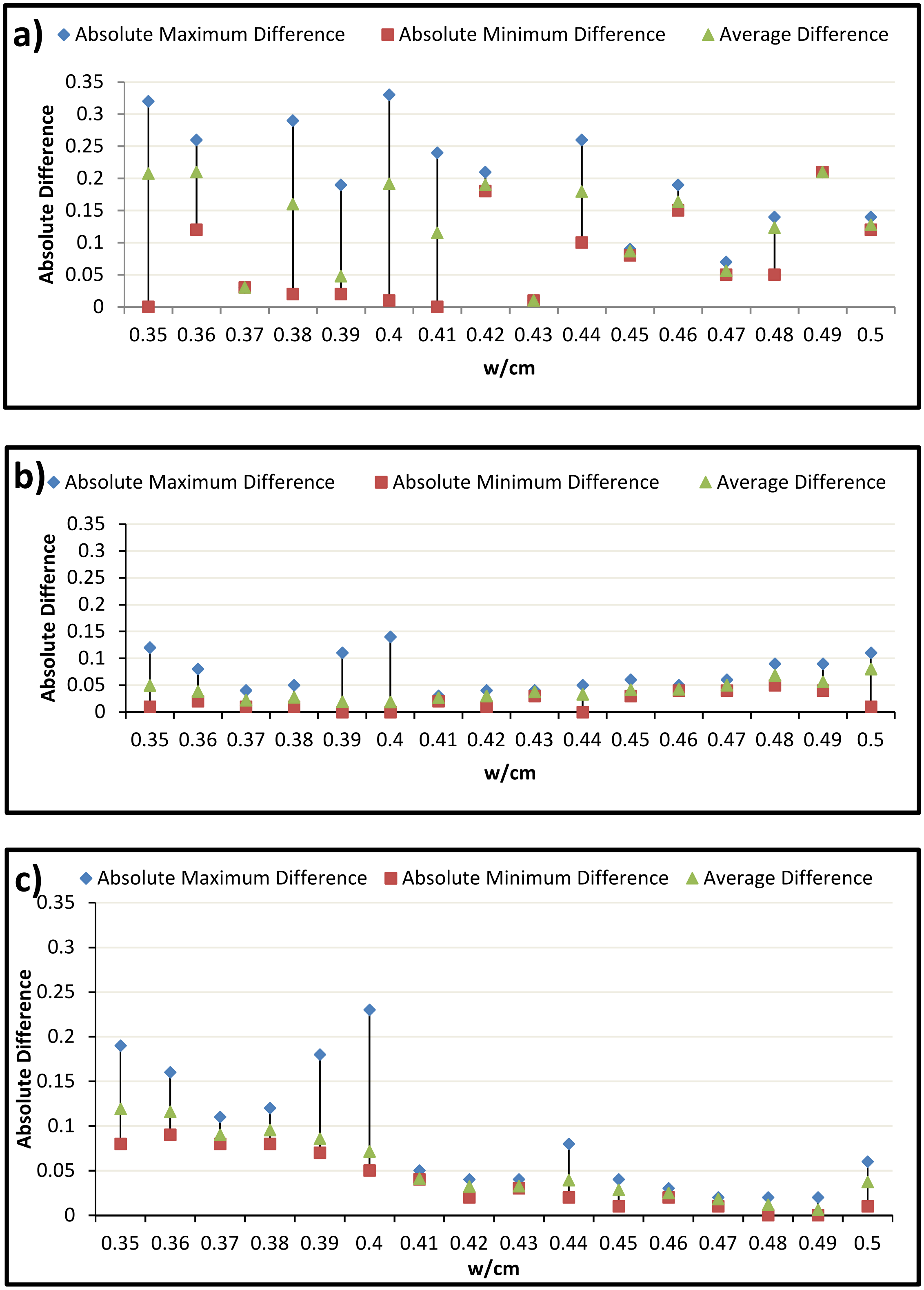


Figure 18 Absolute difference a) User-program b) Type I c) Type III

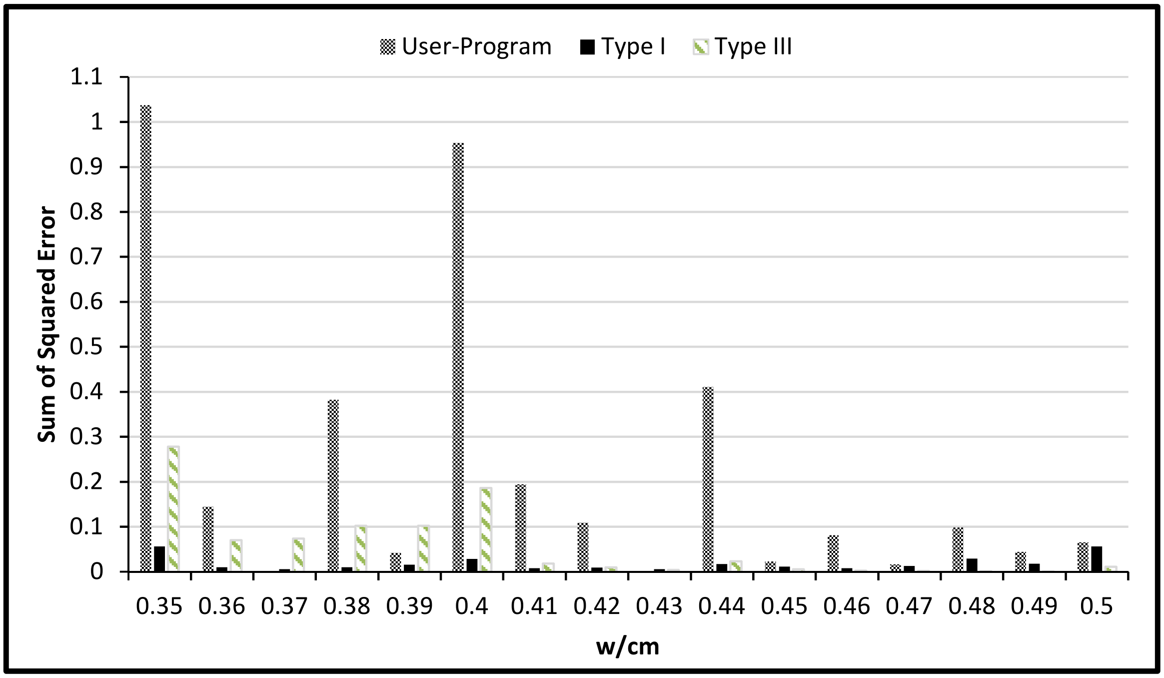
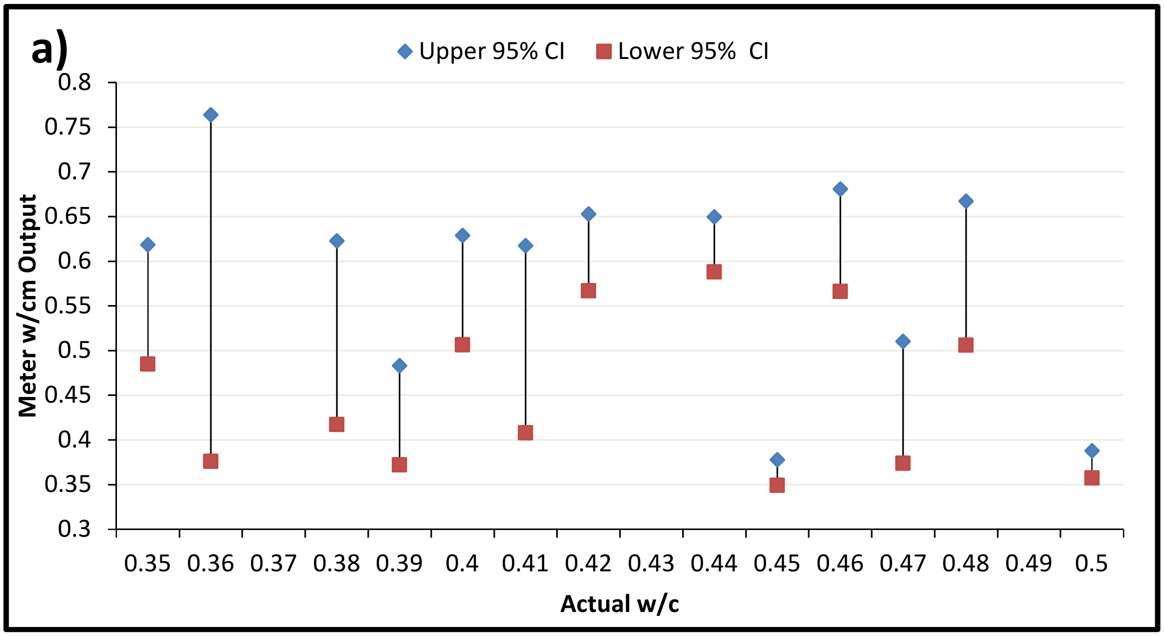


Figure 18 Sum of square error for the three different modes across concrete w/cm mixtures validated.



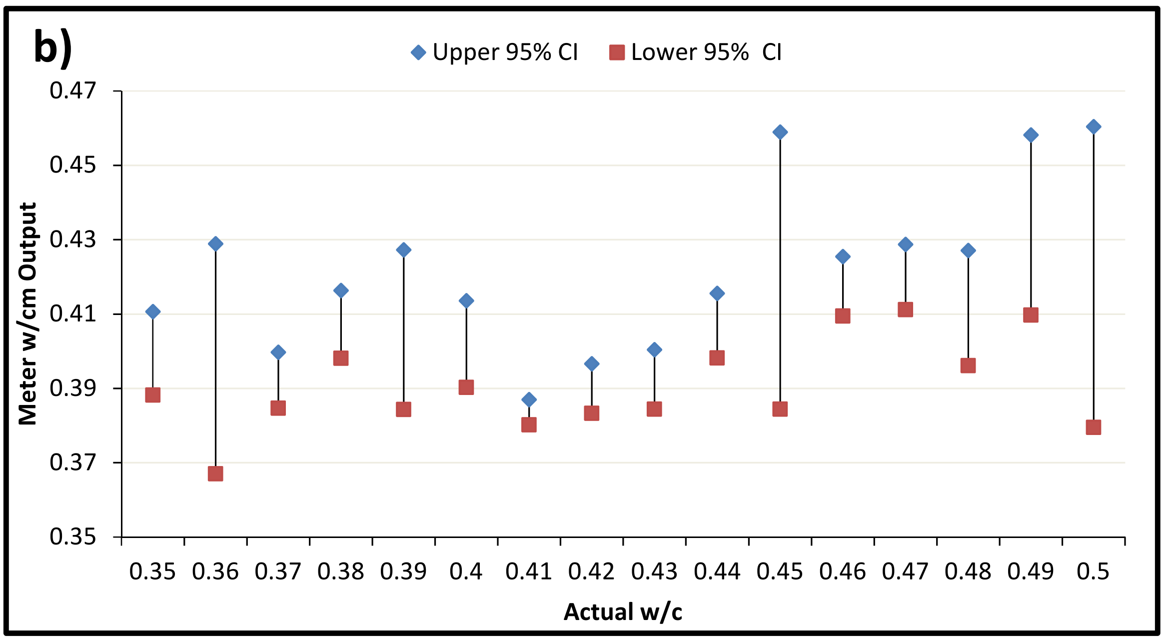


Figure 20 Confidence interval a) User-program, b) Type I and c) Type III

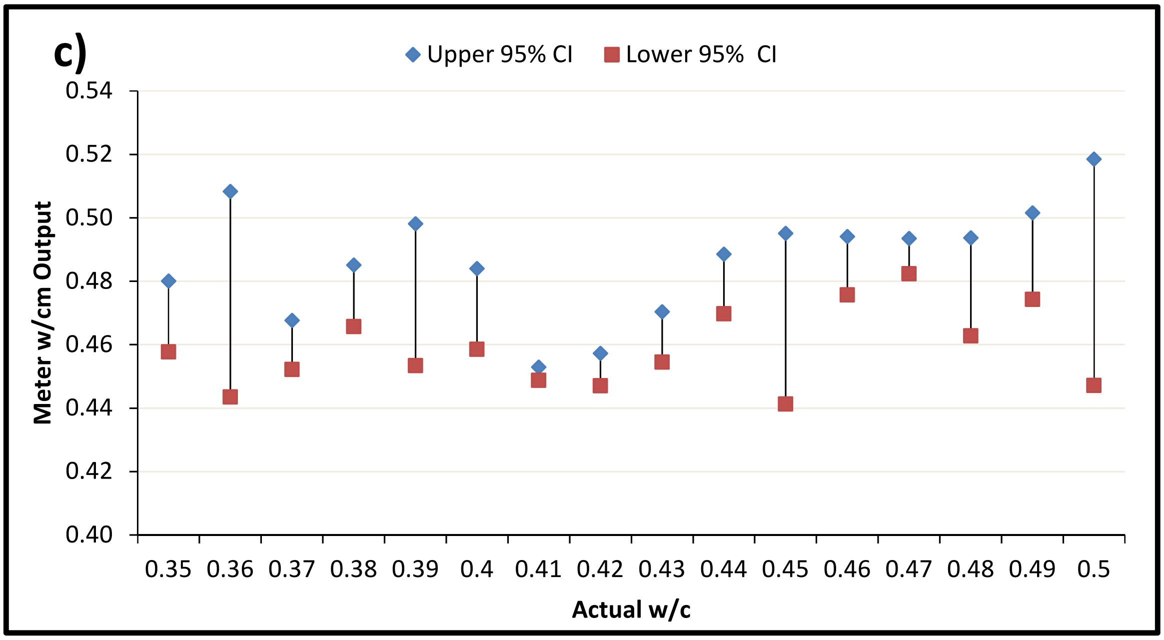


Figure 20 Continued

.

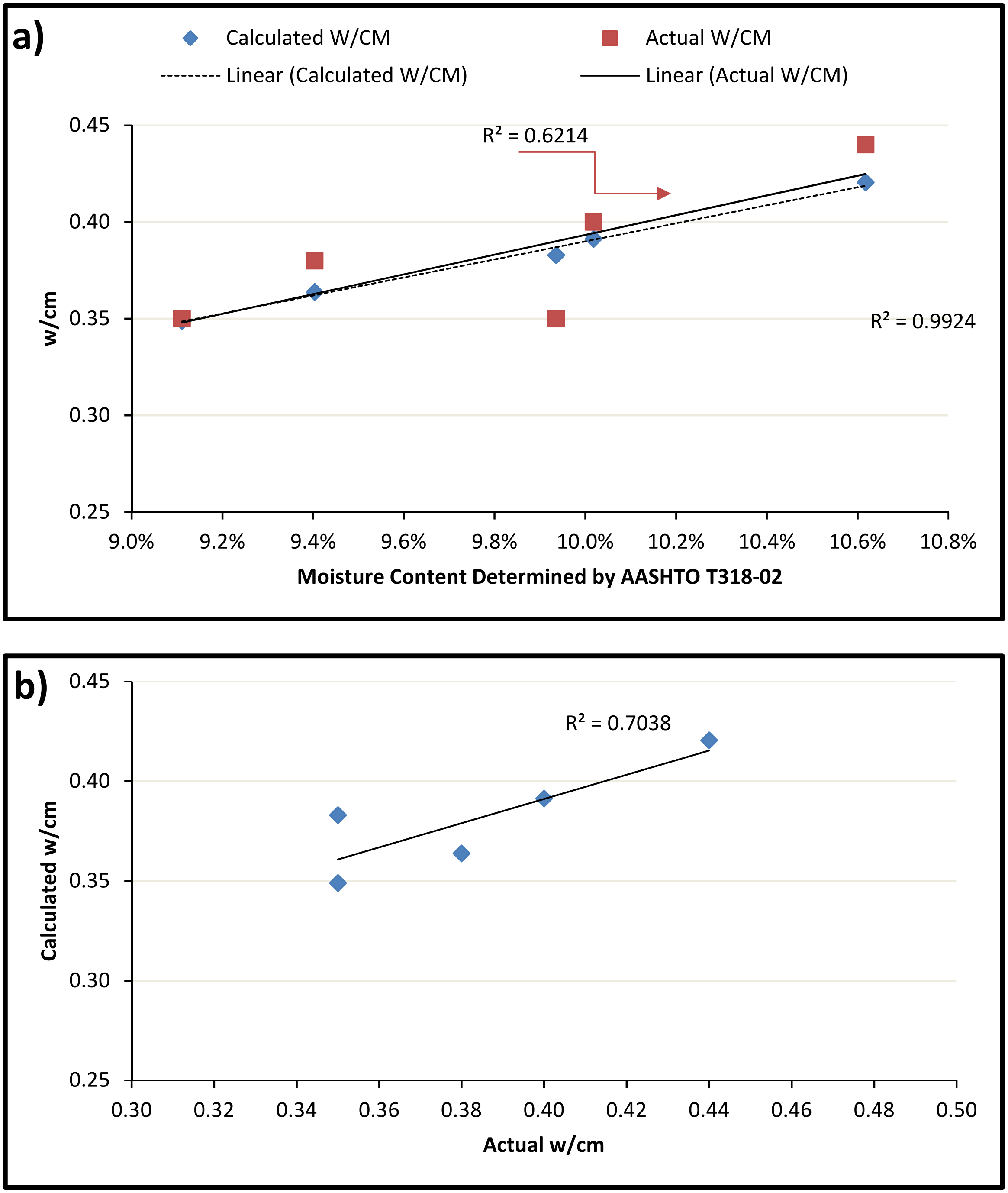


Figure 19 Linear trendline a) Moisture content determined directly from standard b) w/cm calculated from moisture content and mix proportions

CHAPTER 6

CONCLUSION

The Cementometer™ in-situ microwave probe device was verified to produce a good correlation in predicting moisture contents among sand particles, but was not accurate or precise for predicting water amount in cementitious materials. The device produced high variability (high absolute differences and low linear R2 regression correlations) in the predicted w/cm output of concrete samples regardless of the mode selected or the actual w/cm content. Among the modes, Type I and Type III modes had better precision (standard deviation of 0.02) than the user-calibrated program mode (standard deviation of 0.14). One hundred and fifty-seven concrete mixtures were tested ranging from 0.35 to 0.55 w/cm content for validating the meter, among which t-tests indicated that all three modes did not produce accurate w/cm values equivalent to the actual mixture. While Type I and Type III modes produce average w/cm values which may be similar to a typical concrete mixture made in the field, the high variability and a low p-value indicate these modes cannot be used to differentiate between similar but unique concrete mixture w/cm values. The Cementometer™ is not recommended to be used as a quality assurance method for concrete or mortar as it is not precise or accurate enough to differentiate between two similar w/cm contents. The direct reading mode from the Cementometer™ is an acceptable method to assess moisture content in inert sand. The direct reading had a good linear correlation (R^2 of 0.94 to 0.96 for natural or lightweight fines) with the water content. The most precise method to assess moisture content of a mixture was found to be with using the existing AASHTO T 318-02 standard test method. The measurements done to compare moisture content and actual w/cm ratio found a R^2 value of 0.70 for a linear fit, and a p-value of 0.91. It is then recommended to use the AASHTO T 318-02 microwave oven method over the Cementometer™ microwave probe method as an in-situ method which is more precise and accurate to predict the w/cm ratio of a field mixture. Since the direct reading values were also found to be dependent on the temperature of the water, it is suggested that if the user chooses to calibrate their own mixture, the same temperature as would be expected on the day(s) of testing be used for the mix water.

APPENDIX A

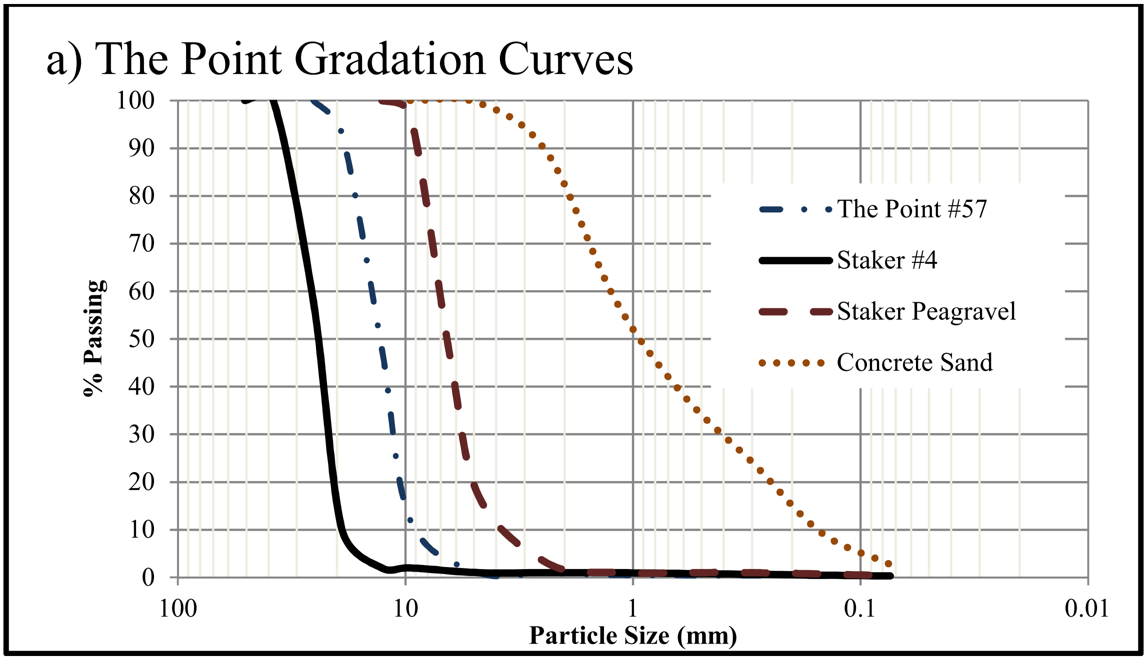
AGGREGATE AND CEMENT PROPERTIES

A.1 Aggregate Properties

Table 9 Properties of Aggregates Used for Concrete Mixtures

In-House			
Aggregate Properties	NMAS	Bulk Specific Gravity SSD	Absorption Capacity
Beck Street Pea Gravel	0.5"	2.67	1.28
Beck Street Limestone Aggregate	0.75"	2.62	0.43
Beck Street Sand	#4	2.57	1.90
Utelite Structural Fines*	#8	1.559	1.22
Harper			
Aggregate Properties	NMAS	Bulk Specific Gravity SSD	Absorption Capacity
#67 Coase Aggregate*	0.75"	2.67	1.06
#8 Coarse Aggregate*	0.5"	2.67	1.06
Natural Sand*	#4	2.65	1.15
The Point			
Aggregate Properties	NMAS	Bulk Specific Gravity SSD	Absorption Capacity
Staker East #57	0.75"	2.50	1.75
Staker West #57	0.75"	2.55	1.40
Staker East #4	1.5"	2.50	1.40
Staker West #4	1.5"	2.55	1.40
Staker East Sand	#4	2.60	1.10
Staker West Sand	#4	2.59	1.40
Staker East Pea gravel	3/8"	2.50	1.90
Staker West Pea gravel	3/8"	2.55	1.90

*Gradation Curve not availabl



East and West pit of The Point aggregates have the same particle distribution.

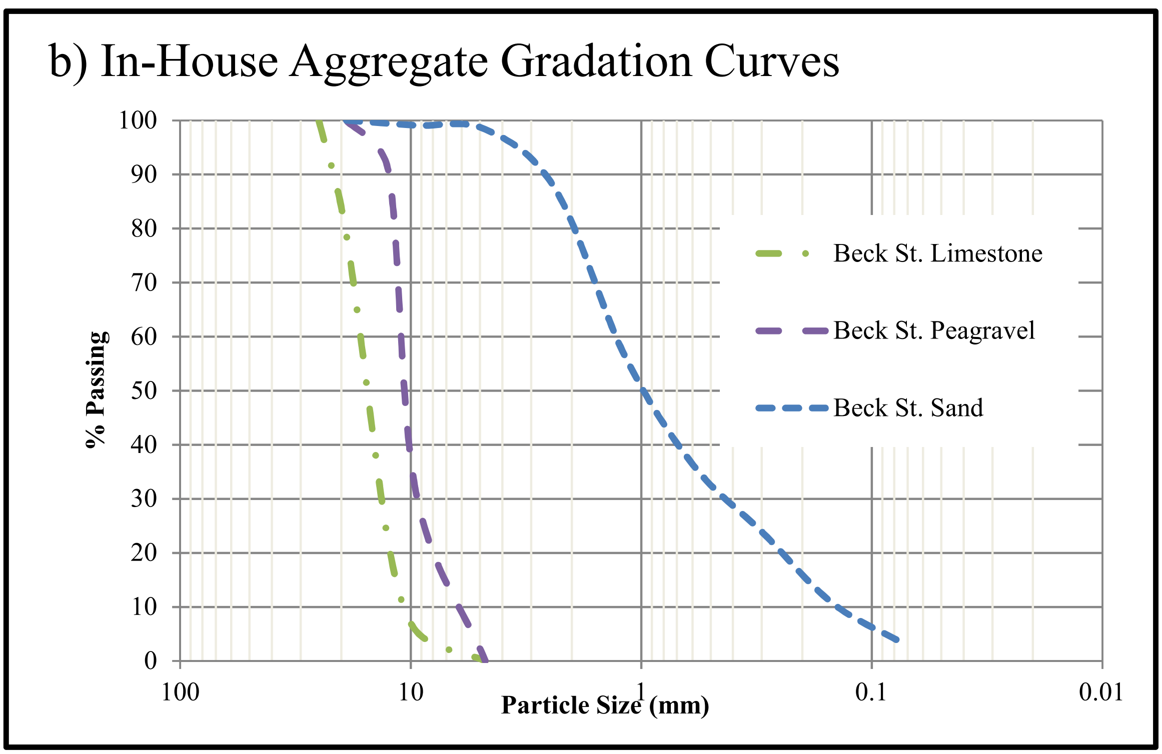


Figure 20 Gradation curves a) The Point mixtures b) In-house mixture

A.2 Cement and FlyAsh Properties

Material: Portland Cement
Type: ASTM C150 Type II-V
Supplier: Lafarge-Holcim Devil's Slide Plant

Table 10 Cement Properties

Chemical Properties (Cement)		
Item	Limit %	Result %
SiO2	-	20.4
Al2O3	6.0 Max	4
Fe2O3	6.0 Max	3.5
CaO	-	63.5
MgO	6.0 Max	2.7
SO3	2.3 Max	3
Loss on Ignition	3.0 Max	2.3
Insoluble Residue	0.75 Max	0.42
CO2	-	1.7
Limestone	5.0 Max	4.3
CaCO3 in Limestone	70 Min	89
Inorganic Processing Addition	5.0 Max	0
Bogue Estimates		
C3S	-	57
C2S	-	15
C3A	5 Max	5
C4AF	-	10
C3S + 4.75C3A	-	80.8
Equivalent Alkalies (%)	0.60 Max	0.55
Physical Properties (Cement)		
Item	Limit	Result
Air Content %	12 Max	7
Blaine Fineness (m2/kg)	260 Min	408
Autoclave Expansion % ASTM C151	0.80 Max	0.04
Initial Vicat (minutes)	45-375	113
Mortar Bar Expansion % ASTM C1038	-	0.01
Heat of Hydration 7 days (kJ/kg)		75
Compressive Strength (psi)		
3 days	1450 Min	4390
7 days	2470 Min	5330

Material: Fly Ash

Type: ASTM C618 Class F
Supplier: Headwaters Resources Navajo

Table 11 FlyAsh Properties

Chemical Properties (Fly Ash)		
Item	Limit %	Result %
SiO2	-	59.35
Al2O3	-	22.45
Fe2O3	-	4.68
Sum of Constituents	70 Min	86.48
SO3	5 Max	0.41
CaO	6.0 Max	5.07
Moisture	3 Max	0.06
Loss on Ignition	6 Max	6
Available Alkalies as Na2O	1.5 Max	1.38
Physical Properties (Fly Ash)		
Item	Limit %	Result %
Fineness, % retained on #325	34 Max	19.91
Water Requirement, % Control	105 Max	95
Autoclave Soundness	0.8 Max	0.01
Density		2.35
Strength Activity Index		
7 day, % of control	75 Min	89
28 day, % of control	75 Min	95

APPENDIX B

PROCEDURE FOR CEMENTOMETER™

Step 1: Place the sand in an oven for 24 hours to dry in order to insure complete evaporation of water from the surface and internal pores.

Step 2: Remove sample from oven and allow sample to cool down.

Step 3: Immerse sample in water for 24 hours to ensure full water saturation.

Step 4: Remove excess water from sample and prepare at SSD in accordance to ASTM C 128-01.

The sand sample is now at SSD condition.

Step 5: Place the coarse aggregate sample in an oven for 24 hours to dry in order to insure complete evaporation of water from the surface and internal pores.

Step 6: Remove sample from oven and allow to cool.

Step 7: Immerse sample in water for 24 hours to ensure full water saturation

Step 8: Remove excess water from the surface of sample in order to have sample at SSD condition.

The coarse aggregate sample is now at SSD condition.

Once the fine and coarse aggregates are conditioned, a proper weight for the mixture is batched and the mixing procedure is ready to start.

Step 9: Spray down inner dome to moisten drum.

Step 10: Remove any excess water and start revolving the mixer.

Step 11: Add 10% of the water and coarse aggregate into the drum.

Step 12: Add 50% of the fine aggregates and cement into the drum.

Step 13: Add 60% of the coarse aggregate and the remaining water until approximately ¼ to 1.3 of the water is remaining in the reservoir being used to contain the water.

Step 14: Add the remaining fine aggregate and cement to the drum followed by the remaining coarse aggregate and water.

Step 15: Let sample mix until a proper paste has been achieved

Step 16: Remove a sample of plastic concrete from the drum in order to take calibration measurements. Once the readings are obtained, return sample to mixer and add more water to reach next w/cm. Repeat steps 7 and 8.

Once an even mixture is attained, the cementitious materials are added with the remaining water. Once a proper paste is achieved in accordance to ASTM C192/C192 M standard, a sample large enough to allow the probes in with sufficient clearance from all directions is removed and placed in a container that allows at least a 2-inch clearance around the meter's probes. After the calibration readings are obtained, the sample is returned to the mixer and additional water is added to achieve a 0.05 increase in w/cm. The addition of water procedure was repeated 9 times until the full range of calibration values were obtained

APPENDIX C

AASHTO MICROWAVE TEST SAMPLE CALCULATIONS

C.1 Equations

Absorption Capacity (AC) = $\frac{W_{SSD-}W_{OD}}{W_{OD}} \times 100\%$

where:

W_{SSD} = Saturated surface dry weight of the aggregate

W_{OD} = Oven dried weight of the aggregate

Or alternatively:

$$W_{SSD} = \frac{(AC+1)\,W_{OD}}{100\%}$$

where:

Water Absorbed by Aggregates = $W_{SSD} - W_{OD}$

Free Water = Original Amount Added – Water Absorbed by Aggregates

Actual w/cm = [free water] / total cement

Input w/cm = [free water + absorbed water] / total cement

= Actual w/cm – [absorbed water]/total cement

$$Volumetric\ Ratio = \frac{Volume_{Water}}{Volume_{Solids}}$$

Where:

$$Volume_{Water} = \frac{Mass\ of\ Water}{Density\ of\ Water}$$

$$Volume_{Solids} = Volume_{CementitiousMaterial} + Volume_{CoarseAggregate} + Volume_{Sand}$$

C.2 AASHTO T318-02 Microwave Test

Water-Cement Ratio Calculation

Repeated from the standard:

$$WC = \frac{W_{tray+cloth+original\ sample} - W_{tray+cloth+after\ microwaved\ sample}}{W_{tray+cloth+original\ sample} - W_{tray+cloth}} * 100\%$$

$WT = WC * UW_{concrete}$

Assumptions:

- Weight of tray and cloth combined was tarred so as not to contribute to the weight measured of the original sample or after being microwaved.
- The original sample obtained for the microwave test is representative of the entire concrete mixture.
- All batch weights are reported for SSD condition of aggregates.
- Total water measured by the microwave method includes batched water, water that was absorbed into aggregates to make it SSD condition, plus any additional water added before hardening.

The following calculations can then be made:

Water Content WC = (3.895 – 3.505) / (3.895) *100 = **10.02%**

Total Water WT = (10.02%*144.8*27) = **392 pcy**

Table 12 Mixture Proportions in AASHTO Microwave Test

Mixture Proportions (SSD condition)		
	AC	Batch Weights (lb)
Cement		19.7
Fly Ash		3.9
Sand	1.9%	25.65
Coarse Agg1	0.43%	9.9
Coarse Agg2	0.28%	29.7
Water		9.44
Concrete Unit Weight (lb/cf)	144.8	
Original Sample (lb)	3.895	
Sample After Microwaved (lb)	3.505	

Total batch weights $\sum W_{each\ material\ as\ batched}$= (19.7+3.9+25.65+9.9+29.7+9.44) = **98.29 lb**

Total cementitious $W_{cementitious}$= (19.7+3.9) = **23.6 lb**

Water absorbed by aggregate

Sand	1.9%*25.65 = 0.4874 lb
Coarse Agg1	0.43%*9.9 = 0.0426 lb
Coarse Agg2	0.28%*29.7 = 0.0832 lb
$\sum(AC_i * W_{aggi})$	= **0.6131 lb**

Water in sample $W_{water\ in\ sample}$ = (3.895 – 3.505) = **0.39 lb**

Scale Factor *SF* = (0.39) / (98.29) = **0.0396**

Actual w/cm ratio = (9.44) / (23.6) **= 0.40**

Calculated w/cm ratio = (0.39 – 0.0243*0.0396) / (0.0396*23.6) = **0.39**

APPENDIX D

STATISTICS

D.1 Absolute Difference of Calibration Data to Actual w/cm

Difference of any given $\frac{\mathrm{w}}{\mathrm{cm}}$ mode output value to Actual $\frac{\mathrm{w}}{\mathrm{cm}}$: $\mathbf{x}_{\boldsymbol{i,mode}}$

$$= \boldsymbol{w/c_{Actual}} - \boldsymbol{w/c_{i,mode}}$$

Note if the mode output is "out of range" these calculations will be omitted.

Absolute Maximum Difference for a mode output value at a given w/cm: $\boldsymbol{D_{max}} =$

$$\max_{\boldsymbol{given\ w/c}} \left|\mathbf{x}_{\boldsymbol{i,mode}}\right|$$

Absolute Minimum Difference for a mode output value at a given w/cm: $\boldsymbol{D_{min}}$

$$= \min_{\boldsymbol{given\ w/c}} \left|\mathbf{x}_{\boldsymbol{i,mode}}\right|$$

Average Absolute Difference for a mode output value at a given w

$$/\mathrm{cm}\text{: } \boldsymbol{D_{ave}} \quad \frac{\sum\left|\mathbf{x}_{\boldsymbol{i,mode}}\right|}{\boldsymbol{N_{mode,given\ w/c}}}$$

where $N_{mode,given\ w/c}$ = number of readings corresponding to that Actual w/cm not including any "out of range" values.

Square Error $= \left(\mathrm{x}_{i,mode}\right)^2$

Sum of Square Error $\mathbf{SSE} = \sum \left(\mathbf{x}_{\boldsymbol{i,mode}}\right)^{\mathbf{2}}$

For this data set of 0.35 w/cm ratios, the following can be calculated:

D_{max} = **0.32**

D_{min} = **0.00**

D_{ave}= **0.2072**

(again note, the OOR reading is not included in the average difference)

SSE =

0.0025+0.1024+0.0625+0.0729+0.0841+0.09+0.0004+0.00+0.0841+0.0676+0.00+0.0001+0.1024+0.0729+0.900+0.0529+0.090+0.0625

= 1.0373

Table 13 Sample Calculation for User-Program Mode Value Measurements at a 0.35 w/cm Actual Content

Actual w/c_{Actual}	User-Program $w/c_{i,User}$	Difference $x_{i,User}$	Absolute Difference $\lvert x_{i,User} \rvert$	Square Error $(x_{i,mode})^2$
0.35	0.30	0.05	0.05	0.0025
0.35	0.67	-0.32	0.32	0.1024
0.35	0.60	-0.25	0.25	0.0625
0.35	0.62	-0.27	0.27	0.0729
0.35	0.64	-0.29	0.29	0.0841
0.35	OOR			
0.35	0.65	-0.3	0.3	0.09
0.35	0.37	-0.02	0.02	0.0004
0.35	0.35	0	0	0
0.35	0.64	-0.29	0.29	0.0841
0.35	0.61	-0.26	0.26	0.0676
0.35	0.35	0	0	0
0.35	0.36	-0.01	0.01	0.0001
0.35	0.67	-0.32	0.32	0.1024
0.35	0.62	-0.27	0.27	0.0729
0.35	0.65	-0.3	0.3	0.09
0.35	0.58	-0.23	0.23	0.0529
0.35	0.65	-0.3	0.3	0.09
0.35	0.60	-0.25	0.25	0.0625

REFERENCES

American Association of State and Highway Transportation Officials (2015). "AASHTO T 318-02 Standard Method of Test for Water Content of Freshly Mixed Concrete Using Microwave Oven Drying." American Association of State and Highway Transportation Officials, Washington, D.C.

Anderson, C. (2006). "Determining the Complex Permittivity of Materials with the Waveguide-Cutoff Method," thesis, presented to Baylor University at Waco, TX, in partial fulfillment of the requirements for the degree of Masters of Science.

American Society for Testing and Materials (ASTM) (2012a). "ASTM C150/C150M-12 Standard Specification for Portland Cement." American Society for Testing and Materials, West Conshohocken, PA.

American Society for Testing and Materials (ASTM) (2012b). "ASTM C618-12 Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete". American Society for Testing and Materials , West Conshohocken, PA.

American Society for Testing and Materials (ASTM) (2012c). "ASTM C127-12 Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Coarse Aggregate". American Society for Testing and Materials, West Conshohocken, PA.

American Society for Testing and Materials (ASTM) (2015). "ASTM C128-15 Standard Test Method for Relative Density (Specific Gravity) and Absorption of Fine Aggregate". American Society for Testing and Materials, West Conshohocken, PA.

Baker-Jarvis, J., Janezic, M., & DeGroot, D. C. (2010). "High-frequency dielectric measurements." *Instrumentation & Measurement Magazine (IEEE),* 13(2), 24-31. DOI 10.1109/MIM.2010.5438334

Blonquist, J. M., Jones, S. B., Lebron, I., & Robinson, D. A. (2006). "Microstructural and phase configurational effects determining water content: Dielectric relationships o aggregated porous media." *Water Resources Research*, 42(5). DOI:10.1029/2005WR004418

Chang, K. (2005). *Encyclopedia of RF and Microwave Engineering*. John Wiley, Hoboken, NJ.

Chen, W., Shen, P., & Shui, Z. (2012). "Determination of water content in fresh concrete mix based on relative dielectric constant measurement." *Construction and Building Materials*, 34, 306-312. DOI:10.1016/j.conbuildmat.2012.02.073

Clipper Controls Inc. (2017). "Dielectric Constant Values." http://www.clippercontrols.com/pages/Dielectric-Constant-Values.html. (Jan. 1. 2017)

Feynman, R. P., Leighton, R. B., & Sands, M. (2013). "The Feynman lectures on physics." Online lecture.

Gadani, D. H., & Vyas, A. D. (2008). "Measurement of complex dielectric constants of soils of Gujarat at X- and C-band microwave frequencies." *Indian Journal of Radio & Space Physics*, 37, 221-229. Retrieved from http://www.researchgate.net

James Instruments (2010). "Cementometer™ Hand Held Microwave Moisture Meter For Water/Cement Ratios of Fresh Concrete". http://www.ndtjames.com/Cementometer-p/t-c.html.

Kim, Y., Lee, K., Bang, J., & Kwon, S. (2014). "Effect of W/CM ratio on durability and porosity in cement mortar with constant cement amount." *Advances in Materials Science and Engineering*, 1-11. Retrieved from http://dx.doi.org/10.1155/2014/273460

Makul, N. (2013). "Dielectric permittivity of various cement-based materials during the first 24 hours hydration." *Open Journal of Inorganic Non-metallic Materials*, 03(04),53-57. Retrieved from http://dx.doi.org/10.4236/ojinm.2013.34009

Naik, T. R. & Ramme, B. W. (1987) "Determination of the water content of concrete by the microwave method." *Cement and Concrete Research.* 17(6), 927-938. doi:10.1016/0008-8846(87)90081-0

Nave, C. R. (2012). "HyperPhysics; Polarization of Dielectric." http://hyperphysics.phy-astr.gsu.edu/hbase/electric/dielec.html

Peterson, K. W. & Sutter, L. L. (2011). "Impact of Hydrated Cement Paste Quality and Entrained Air-Void System on the Durability of Concrete" *Report RC-1552H-363852-7*, Michigan Department of Transportation, Houghton, MI.

Pozar, D. M. (2012). *Microwave Engineering*. John Wiley, Hoboken, NJ.

Salgado, R. (2008). *The Engineering of Foundations*. McGraw-Hill, Boston, MA.

Shen, P., Lu, L., He, Y., Wang, F., & Hu, S. (2016). "Hydration monitoring and strength prediction of cement-based materials based on the dielectric properties."

Construction and Building Materials. (126), 179-189. Retrieved from http://dx.doi.org/10.1016/j.conbuildmat.2016.09.030

Wang, J. R., & Schmugge, T. (1980). "The dielectric properties of soil-water mixtures at microwave frequencies." *Radio Science*. 15(5), 977-985.

Wikipedia. (2006, November). *Parallel Plate Capacitor*